

INTERLABORATORY
STUDY 93-3
AUGUST 1993

POLYCHLORINATED BIPHENYL (PCB) ISOMERS

IN SUPPORT OF

THE INTEGRATED
ATMOSPHERIC
DEPOSITION
NETWORK
(IADN)

NOVEMBER 1994

QD
139
P53
C87
1993
MOE
LOAN



Environment
Canada

Environnement
Canada

Atmospheric
Environment
Service

Service
de l'environnement
atmosphérique

Copyright Provisions and Restrictions on Copying:

This Ontario Ministry of the Environment work is protected by Crown copyright (unless otherwise indicated), which is held by the Queen's Printer for Ontario. It may be reproduced for non-commercial purposes if credit is given and Crown copyright is acknowledged.

It may not be reproduced, in all or in part, for any commercial purpose except under a licence from the Queen's Printer for Ontario.

For information on reproducing Government of Ontario works, please contact ServiceOntario Publications at copyright@ontario.ca

ISBN 0-7778-3391-3

INTERLABORATORY STUDY 93-3
POLYCHLORINATED BIPHENYL (PCB) ISOMERS
IN SUPPORT OF
THE INTEGRATED ATMOSPHERIC DEPOSITION NETWORK (IADN)
AUGUST 1993

Report Prepared by

Sylvia Cussion

for

**Quality Management Unit
Laboratory Services Branch
Ontario Ministry of Environment and Energy
PIBS: 3299E**

and

**Air Quality Research Branch
Atmospheric Environment Service
Environment Canada
CARD Report: 94-010**



Environment Ontario
Laboratory Library
125 Resources Rd.
Etobicoke, Ontario M9P 3V6
Canada

The author gratefully acknowledges and thanks Sathi Selliah and George Steinke for the preparation of materials, and Renata Baily, Syed Iqbal, and Maris Lusi for their help in reviewing the results.

TABLE OF CONTENTS

1	SUMMARY OF INTERLABORATORY STUDY 93-3	Page 1
2	INTRODUCTION	Page 2
3	PROCEDURE	Page 3
	3.1 Preparation of Ampouled Standards	Page 3
	3.2 Sample Distribution	Page 3
	3.3 Analytical Methodology	Page 3
	3.4 Data Reporting	Page 3
4	DISCUSSION	Page 4
5	CONCLUSION	Page 12
6	REFERENCES	Page 12
7	APPENDIX 1 - RESULTS AND GRAPHS	Page 13
8	APPENDIX 2 - PARTICIPANTS AND CORRESPONDENCE	Page 58

1 SUMMARY OF INTERLABORATORY STUDY 93-3

Interlaboratory Study 93-3 was the second study for Polychlorinated Biphenyl (PCB) isomer analysis initiated in support of the Integrated Atmospheric Deposition Network (IADN). It provides an assessment of between-laboratory variability for laboratories analyzing PCB isomers in precipitation and/or ambient air. Participation was limited to laboratories which contribute to the IADN database or related programs. This study was sponsored by the Canada-Ontario Agreement (COA) Air Toxics Workgroup, and conducted as a joint project between the Atmospheric Environment Service (AES) of Environment Canada and the Quality Management Unit (QMU), Laboratory Services Branch (LSB) of the Ontario Ministry of Environment and Energy (MOEE).

Six laboratories received a set of four ampouled standards. Results were received from five of the laboratories. The parameter list consisted of 48 different PCB isomers. Ampoules 1 and 2 were for direct injection instrumental analysis. Ampoules 3 and 4 required analytical Clean-up prior to instrumental analysis.

The results from this study demonstrate an interlaboratory mean and median that agree with the target within $\pm 15\%$ for the majority of the PCB isomers in this study, and within $\pm 30\%$ of the target for the remainder. This is similar to the first study⁴ among these participating laboratories. However the between-laboratory variability has not improved from the first study, with most isomers having a %RSD in the range 20-40%. This is larger than is considered an acceptable degree of variability in a similar interlaboratory study⁵. Problems with calibration appear to be the main source of this variability, though non-detection of some isomers also contributes to this variability. Two participants had only 26.4% and 47.7% of their results within 75-110% of the target, while the remaining participants had greater than 66% of their results in the range 75-110% of the target. These results re-emphasize the need for a common reference standard to be used by all of the laboratories contributing to the IADN database so as to avoid biases in long term data sets.

The results from the solutions processed through analytical Clean-up also demonstrated good agreement with the target for the interlaboratory mean and median. However the between-laboratory variability was again greater than 20%. Some individual laboratories demonstrated some problems with their Clean-up procedure, primarily some degree of loss of the target compounds, though it varied from isomer to isomer. Contamination of materials used in the Clean-up procedure by some of the participants may also have contributed to this variability.

2 INTRODUCTION

Interlaboratory performance studies are conducted to assess the comparability and accuracy of data among different laboratories. These studies are useful for the identification of biases, precision and accuracy problems. Participation in such studies can serve as a guide for improving individual laboratory performance and maintaining performance standards.

This study was the second in a series designed to assess the analytical variability among laboratories contributing to the Integrated Atmospheric Deposition Network (IADN). IADN was established as a joint venture between Canada and the United States under the direction of the International Joint Commission¹. The intent of IADN is to identify toxic airborne substances in the Great Lakes Basin, and by means of the network, quantify the total and net atmospheric loadings of these contaminants, and define spatial and temporal trends in the atmospheric deposition of these substances. Data from several participating agencies is to be merged into a central database. Comparability of these contributing data sets is an important component of the IADN Quality Assurance Implementation Plan². This interlaboratory study for organics analysis, provides information on the laboratory component of between agency differences, and can be used to help establish the comparability of the data sets. It is a recommended activity of the IADN Quality Assurance Program Plan³. Sponsorship of this interlaboratory study was through the Canada-Ontario Agreement (COA) Air Toxics Workgroup. Funding for the purchase of materials and co-ordination of this study came from the Atmospheric Environment Service (AES) of Environment Canada. Sample preparation for this study was done by the Quality Management Unit (QMU) of Laboratory Services Branch (LSB) of the Ontario Ministry of Environment and Energy (MOEE).

Interlaboratory Study 93-3 targets laboratories analyzing for Polychlorinated Biphenyl (PCB) isomers in precipitation and/or ambient air. From the proposed list of 51 PCB isomers, a final target list of 48 PCB isomers was chosen for this study, using available solutions from the "PCB suite" recommended as the IADN target list³. Two additional isomers, PCB194 and PCB195 that are not part of the "PCB suite" were also added. The aim of this study was to continue to monitor the comparability among the participating laboratories. Two aspects of PCB analysis were addressed in this study: comparability of instrumental calibration and analytical Clean-up of interferences. Two ampouled standards ready for direct instrumental analysis and containing all 48 PCB's on the target list, were provided to assess calibration. Two additional ampoules containing an interferant and therefore requiring Clean-up, were also provided to the participants.

A list of participants is given in Appendix 2. Each participant was assigned a unique identification code for ease in data manipulation.

Section 3 describes sample preparation, sample distribution, analytical methodology, and data evaluation procedures. Final results are tabled in Appendix 1 and discussed in Section 4.

3 PROCEDURE

3.1 Preparation of Ampouled Standards

Neat PCB isomers of 99% + purity were purchased from Ultra Scientific and AccuStandard by AES. All subsequent work was done by the QMU of LSB, MOEE. Concentrated stock solutions of each isomer were prepared in toluene and sealed into 5 mL amber ampoules. The stock concentrations were between 10 to 12 mg/L and verified using gas chromatography/mass spectrometry analysis by an analytical unit at LSB not involved in analysis of ambient air or precipitation. Ampouled solutions were stored in a freezer at -20°C.

Solutions for the interlaboratory study were prepared from the concentrated stock solutions by diluting appropriate aliquots into a combined solution. All combined solutions for the study were prepared in iso-octane. Concentration levels were designed to fall in the routine analytical range of most participants. The first two solutions were sealed in 5 mL amber ampoules and labelled 93-3-PCB1 and 93-3-PCB2. The second two solutions were prepared at the same concentration as the first two, with the addition of an aliquot of vegetable oil at the same concentration range as the PCB's. These solutions were also sealed in 5 mL amber ampoules and labelled 93-3-PCB3 and 93-3-PCB4. All ampoules were stored in a freezer at -20°C until shipped to the participants.

3.2 Sample Distribution

Samples were packed into styrofoam shipping containers and shipped by Purolator Courier to the participating laboratories. A list of the laboratories receiving sample sets is given in Appendix 2. Samples were shipped on August 18, 1993. A copy of all correspondence is also included in Appendix 2.

3.3 Analytical Methodology

Participating laboratories were requested to analyze the samples using their routine in-house methods used to analyze precipitation and/or ambient air samples for the IADN program. Two solutions (93-3-PCB1 and 93-3-PCB2) were ready for direct instrumental injection and participants were asked not to do any sample preparation steps. Participants were specifically asked to process ampoules 93-3-PCB3 and 93-3-PCB4 through their routine analytical Clean-up procedure prior to instrumental analysis. Participants were requested on the report form provided (Appendix 2) to summarize their Instrument, Detector and GC column used for the analysis, as well as Clean-up conditions. All participants were assigned a unique identification code that does not correspond to the order the participants are listed in Appendix 2.

3.4 Data Reporting

Results were submitted to AES in written form. All data were manually entered by laboratory code into an electronic spreadsheet.

The participating laboratories were mailed a copy of the tables of results on June 20, 1994. Laboratory 9332 indicated that they had missed adding the remark code "c" for PCB110 in ampoules 93-3-PCB3 and 93-3-PCB4. They also identified a typographical error for their result for PCB114 in ampoule

93-3-PCB4. They had reported a value of 10.52 and a value of 10.42 had been entered into Table 4. This correction has been made in this report.

The interlaboratory mean, median, standard deviation (SD), and relative standard deviation (%RSD) were calculated for each isomer in each ampoule for which there were 2 or more laboratories reporting results and included in Tables 1-4, Appendix 1. As detection limits were not provided by all of the participants, results that were reported as "ND" were assigned a value of "0" for the purposes of performing the calculations. As the data set is small, these calculated values are provided as an approximate indicator of the spread of the data and may not necessarily be statistically correct. The instrumentation and analytical clean-up information is provided in Table 5. Table 6 lists the distribution of each participant's results relative to the target for the injection-ready solutions and the ampoules processed through Clean-up. Tables 7 and 8 are the results for the ampoules processed through Clean-up (93-3-PCB3 and 93-3-PCB4) converted to percent recovery of the target.

4 DISCUSSION

OVERVIEW OF INTERLABORATORY PERFORMANCE

The data report form submitted with the samples listed the 51 PCB isomers that had been part of the proposed list in the invitation to participate in the study. However only 48 of these isomers were in the ampoules, as the majority of the laboratories had indicated that 3 isomers were not on their target list. One laboratory reported a positive result for PCB75, even though it was not present in the ampoules. Only one laboratory had PCB169 on their target list and only two laboratories had PCB126 on their target lists.

For the injection-ready solutions (ampoules 93-3-PCB1 and 93-3-PCB2), approximately 75% of the interlaboratory means and medians were within 85-115% of the target. Almost all of the other means and medians were within 30% of the target. Problem isomers were PCB5 and PCB8, with means that were higher than the target in both ampoules by more than 30% (PCB5: 148.6% and 138.5%; PCB8: 143.1% and 131.5%). This appears to be particularly affected by the high bias of Laboratory 9335 for these two isomers (Tables 1 and 2). Laboratory 9334B also noted a high bias in their results for PCB8. The most likely source of these problems is the co-elution of these two isomers for some of the laboratories. The interlaboratory mean and median were greater than 30% of the target for PCB37 (135.5%) in ampoule 93-3-PCB1. This appears primarily due to the high results from Laboratories 9335 and 9336, both of whom had co-elution of PCB37 with PCB42. This effect was not evident in ampoule 93-3-PCB2, as the interlaboratory mean and median were within 15% of the target. The only isomer with a mean less than 30% of the target was PCB53 (68.9%) in ampoule 93-3-PCB1. This low value appears affected by both the "ND" reported by Laboratory 9334A and the low result reported by Laboratory 9332 (Table 1).

Despite the generally good agreement of the means and medians with the target in ampoules 93-3-PCB1 and 93-3-PCB2, the between laboratory variability is quite high, as demonstrated by the large values for the %RSD. This is particularly pronounced at the lower concentration level of ampoule 93-3-PCB1, for which only nine isomers (PCB28, PCB33, PCB44, PCB52, PCB61, PCB70, PCB114, PCB153 and PCB194) had a %RSD less than 20% (Table 1). At the higher concentration level of ampoule

93-3-PCB2, three isomers had a %RSD of less than 10%, and an additional eleven isomers had a %RSD between 10-20% (Table 2). Unfortunately this does not reflect an improvement from the previous interlaboratory study among this group of laboratories⁴. In both this and the previous study the %RSD for most isomers is in the range of 20-40%. Problem isomers were PCB4, PCB5, PCB8, PCB53, PCB66, PCB77, PCB138, and PCB195, all with a %RSD greater than 40% in both ampoules 93-3-PCB1 and 93-3-PCB2 (Tables 1 and 2).

The actual concentration of the low level ampoule (93-3-PCB1) ranged from 3.10 to 7.91 ng/mL. In a similar interlaboratory study for PCB isomers, Mes, et al⁵ concluded that interlaboratory variability was acceptable (%RSD <20%) as long as the concentration is greater than 5 pg/ μ L (ng/mL). In this study, only four of the isomers with target levels less than 5 ng/mL (PCB33, PCB61, PCB70 and PCB114), had %RSD less than 20%. Even for the other 17 isomers in ampoule 93-3-PCB1 with target levels greater than 5 ng/mL and the higher concentration ampoule 93-3-PCB2 in this study, only five other isomers (PCB28, PCB44, PCB52, PCB153 and PCB194) had %RSD of less than 20%.

The results from this study re-emphasizes the need for a common reference standard to be used among the laboratories contributing to the IADN database. This would help reduce the potential of a 40% or more bias among data sets contributed by different laboratories who may have an individual bias of 20% or more.

Ampoules 93-3-PCB3 and 93-3-PCB4 required processing through analytical Clean-up prior to instrumental analysis. Concentrations levels of the PCB isomers were the same as in ampoules 93-3-PCB1 and 93-3-PCB2, respectively. The interlaboratory mean and median from ampoules 93-3-PCB3 and 93-3-PCB4 do not demonstrate as good agreement with the target as did ampoules 93-3-PCB1 and 93-3-PCB2. Only 50% of the means and medians for ampoules 93-3-PCB3 and 93-3-PCB4 were within 85-115% of the target. Almost all of the remaining isomers were within $\pm 30\%$ of the target.

To aid in evaluating the results, a graphical technique was used for those isomers with 6 or 7 results. As each isomer had a "pair" of results, either Ampoule 93-3-PCB1 and 93-3-PCB2 (injection-ready ampoules), or 93-3-PCB3 and 93-3-PCB4 (ampoules for Clean-up), these results may be plotted on an X-Y plot using the Youden technique⁶. The result from Ampoule 1 or 3 is plotted on the vertical axis and the result from Ampoule 2 or 4 is plotted on the horizontal axis. The interlaboratory mean for each ampoule is also plotted.

The graphs are divided into four quadrants, with the intersection point at the target values. The data points should cluster around the target if random error is the only source of variability. Results in the upper right quadrant are considered biased high and those in the lower left quadrant are biased low. The main source of this type of variability is a difference in analytical standards or inadequate calibration practices. Data points that fall in the lower right or upper left quadrants are considered erratic or out-of-control. Sources of this type of error are more difficult to ascertain. Sources of erratic performance for the injection-ready ampoules could be poor sample injection into the gas chromatograph, a septum leak, poor chromatography if contamination remained from a previous sample, or other instrumental problems. These potential sources of error may be compounded in the ampoules that undergo Clean-up steps due to sample loss or contamination from air pockets in the Clean-up column, contaminated

packing material or solvents in the Clean-up column, poor solvent flow, errors in fractionation or losses during the final evaporation step.

Within-laboratory precision may be assessed by drawing a line between the origin and the intersection of the target values. The closer the data point is to this diagonal line, the better the within-laboratory precision.

The Youden plots for 29 of the PCB isomers in this study are found in Appendix 1, Figures 1 - 58. The graphs for the injection-ready solutions (odd-numbered figures, top graph on each page) all demonstrate a spread between the laboratories in the upper right and lower left quadrants, typically signifying a difference in standards or inadequate calibration practices. The within-laboratory precision is very good, with the majority of data points on or very close to the diagonal line. Some individual outliers are discussed in the individual laboratory review below. These graphs re-emphasize the need for a common reference standard among these laboratories, as noted above.

The Clean-up process (even-numbered figures, lower graph on each page) demonstrates different effects on different isomers. For many isomers, there is no difference in laboratory performance. This can be seen for PCB84 (Fig. 33 & 34), PCB114 (Fig. 41 & 42), PCB136 (Fig. 45 & 46), PCB180 (Fig. 53 & 54) and PCB195 (Fig. 57 & 58). One effect is a greater spread in the results from the laboratories, with laboratories having results either lower or higher than the target than when directly injected. This is demonstrated by PCB4 (Fig. 1 & 2), PCB8 (Fig. 3 & 4), PCB18 (Fig. 5 & 6), PCB42 (Fig. 15 & 16), PCB44 (Fig. 17 & 18), and PCB70 (Fig. 29 & 30). Another effect is overall lower recovery by all of the participants, resulting in a shift in the cluster of data points to the lower left quadrant. This is demonstrated for PCB28 (Fig. 7 & 8), PCB31 (Fig. 9 & 10), PCB40 (Fig. 13 & 14), PCB77 (Fig. 31 & 32), PCB138 (Fig. 47 & 48), and PCB153 (Fig. 49 & 50).

While the second and third groups of graphs listed above demonstrated changes between performance of the injection-ready solutions and the ampoules processed through Clean-up, in only a few cases were there any statistically significant differences at the $\alpha=0.05$ level. Isomer PCB17 was the only case of a significant difference in the mean of ampoule 93-3-PCB1 vs. 93-3-PCB3. There were no significant differences in the means of ampoule 93-3-PCB2 vs. 93-3-PCB4. At the lower concentration level (ampoule 93-3-PCB1 vs. 93-3-PCB3), there was a significant difference in the variability (F-test at $\alpha=0.05$) for isomers PCB40, PCB44 and PCB194. At the high concentration level (ampoule 93-3-PCB2 vs. 93-3-PCB4), there was a significant difference in the variability for isomers PCB16, PCB61, and PCB194. While this suggests that the Clean-up process does not change the overall performance among the laboratories, the high level of between-laboratory variability as noted above is still a matter of concern. Individual laboratories have some problems with specific isomers that are discussed below.

INDIVIDUAL LABORATORY PERFORMANCE

While the participants in this study also participated in Interlaboratory Study 92-1⁴, the laboratories have been assigned different ID Codes in this study. Therefore the following individual review is based only on the results of this study, with no comparisons to previous studies, so as to maintain laboratory confidentiality.

Laboratory 9331

Laboratory 9331 was unable to report any results for this study.

Laboratory 9332

Laboratory 9332 used a dilution factor of 2 for ampoule 93-3-PCB2. They were biased slightly low relative to the target for the injection-ready solutions (ampoules 93-3-PCB1 and 93-3-PCB2). 48% of their results were in the range 75-90% of the target (Table 6 and Fig. 59). Isomers that appear to have been particularly low were PCB53 (Fig. 25), PCB97 (Fig. 35), PCB118 (Fig. 43), PCB138 (Fig. 47), and PCB180 (Fig. 53). They had excellent within-laboratory precision, as indicated on the Youden plots (Figs. 1-58), with all of their data points either on the diagonal line or very close to it. However the tendency for the low bias demonstrates the need for a common reference standard to be used among the laboratories contributing data to the IADN database so as to help each laboratory maintain accurate calibration standards.

To effect a dilution factor of 2 for ampoule 93-3-PCB4, Laboratory 9332 used only 0.5 cm³ of the sample for the Clean-up step (Table 5). Their results for the ampoules processed through Clean-up indicate some losses are occurring during their procedure. 50% of their results are in the range 60-75% of the target and a further 30% are in the range 75-90% of the target (Table 6 and Fig. 60). Particular problem isomers were PCB53, PCB97, and PCB138, all with recoveries below 50% (Tables 7 and 8). The low recoveries are partially due to the low bias in the standards, as noted above. Comparison to the laboratory's in-house recovery checks should indicate what their routine recoveries are for each isomer, and whether a recovery of slightly less than 100% is the best achievable with their analytical method.

Laboratory 9333

Laboratory 9333 was biased high for many of the isomers in the injection-ready solutions (ampoules 93-3-PCB1 and 93-3-PCB2), with 41% of their results in the range 110-130% and 28% of their results in the range > 130% of the target (Table 6 and Fig. 59). As well as being biased high relative to the target, they also tend to be biased high compared to the other participants (Youden plots, odd-numbered Figures 1-57). However they generally had good within-laboratory precision, as most of their data points on the Youden plots were on or close to the diagonal line.

There is a dramatic shift in their results for the ampoules processed through Clean-up. 49% of their results are less than 60% of the target and another 22% are in the range 60-75% of the target (Table 6 and Fig. 60). These results indicate problems with the analytical Clean-up procedure that need to be investigated. Isomers that particularly demonstrate this shift are PCB28 (Figs. 7 and 8), PCB33 (Figs. 11 and 12), PCB40 (Figs. 13 and 14), PCB42 (Figs. 15 and 16), PCB44 (Figs. 17 and 18), PCB47 (Figs. 19 and 20), PCB49 (Figs. 21 and 22), PCB52 (Figs. 23 and 24), PCB66 (Figs. 27 and 28), PCB70 (Figs. 29 and 30), PCB97 (Figs. 35 and 36), PCB101 (Figs. 37 and 38), PCB105 (Figs. 39 and 40), PCB118 (Figs. 43 and 44), PCB136 (Figs. 45 and 46), PCB153 (Figs. 49 and 50), PCB156 (Figs. 51 and 52), PCB180 (Figs. 53 and 54), and PCB194 (Figs. 55 and 56). Most analytical laboratories have established recovery criteria that allows for a lower recovery limit of 80% (or better). These results suggest a much greater loss than 20%, particularly in light of the high bias of the injection standards.

A contamination problem appears to have occurred for isomer PCB4 in ampoule 93-3-PCB3, as the result was biased very high (recovery of 621%, Table 7). This value was excluded from the statistical calculations, as noted in Table 3. No comments or explanations were received from the laboratory when they received the initial tables of results for review.

Laboratory 9334A, B & C

Laboratory 9334 initially provided two sets of results, using two different Clean-up procedures for ampoules 93-3-PCB3 and 93-3-PCB4, and two different instrumental techniques for all of the ampoules. The results were labelled "A" and "B". Prior to the release of the tables of results to the participants, Laboratory 9334 modified their Clean-up techniques to a single-fraction analysis. As extra solutions were available, they repeated their analysis using their revised method and submitted a third set of results. These were labelled "C". Details of the different procedures are provided in Table 5. In the following discussion, each analytical procedure is discussed separately. An overview follows at the end of the discussion for 9334C.

Laboratory 9334A

Laboratory 9334A had 67% of their results for the injection-ready solutions (ampoules 93-3-PCB1 and 93-3-PCB2) in the range of 75-110% of the target (Table 6 and Fig. 59). They did not detect three isomers that were in the ampoules, PCB53, PCB66 and PCB138. They were biased very high for one isomer, PCB 77 (Fig. 31), relative to both the target and the other participants. Their within-laboratory precision for the injection-ready ampoules was good, with most data points in the Youden plots on or near the diagonal line, except for PCB114 (Fig. 41) and PCB180 (Fig. 53).

Laboratory 9334A had problems for several isomers in the solutions requiring Clean-up. Only 43% of their results for ampoules 93-3-PCB3 and 93-3-PCB4 were in the range 75-110% of the target (Table 6 and Fig. 60). However 27% of their results were in the range of less than 60% of the target, indicating losses of the target compounds. As well as the three isomers not detected in the injection-ready solutions, PCB4, PCB40, and PCB77 were not detected in ampoules 93-3-PCB3 and 93-3-PCB4 (Tables 3 and 4). For isomers PCB31, PCB105, PCB118, and PCB156, they were detected in the high concentration ampoule (93-3-PCB4) but were reported "ND" in the low concentration ampoule (93-3-PCB3). Both of the ampoules were diluted by a factor of 5. As the concentration reported is below the known detection limit and detectable levels were measured for ampoule 93-3-PCB4, it would suggest that ampoule 93-3-PCB3 was over-diluted. The low results in ampoule 93-3-PCB4 compared to the target value suggest over-dilution or under recovery from the Clean-up process. Comparison of these results with in-house spike recovery checks may indicate if these problems are inherent in their method.

Problems with within-laboratory precision were also observed for PCB44 (Fig. 18), PCB70 (Fig. 30), PCB114 (Fig. 42), PCB153 (Fig. 50), PCB180 (Fig. 54), and PCB194 (Fig. 56). In all of these cases, the problems appeared to be with the high concentration ampoule (93-3-PCB4), with the results tending to be biased high. Another isomer that was biased high in both ampoule 93-3-PCB3 and 93-3-PCB4, was PCB52 (Fig. 24). These results suggest some contamination in the Clean-up material or equipment that introduces interferants into the sample. The problems noted indicate that Laboratory 9334A needs to review its Clean-up procedure and carefully monitor their performance.

Laboratory 9334B

Laboratory 9334B used the same calibration standards for this method (GC/MS, see Table 5) as was used for the results reported for "Laboratory 9334A" (GC/ECD). Using the GC/MS method, they had approximately the same percentage of results for the injection-ready ampoules 93-3-PCB1 and 93-3-PCB2 (67%, Table 6 and Fig. 59) in the range 75-110% of the target as for the GC/ECD method. However there is a shift lower, as 52% of the GC/MS results were in the range 75-90% as compared to only 35% of the GC/ECD results being in this range. Their results for PCB8 were high but were flagged as such due to a co-eluting compound not on their target list (Tables 1 and 2). Within-laboratory precision was good for the injection-ready ampoules, with the data points on the Youden plots on or close to the diagonal line, except for PCB18 (Fig. 5) and PCB97 (Fig. 35).

Almost 82% of the results for the ampoules processed through Clean-up (93-3-PCB3 and 93-3-PCB4) were in the range 75-110% of the target (Table 6 and Fig. 60). It is not clear why agreement with the target is better after the additional (Clean-up) analytical procedure than when the solutions were directly injected into the instrument. There was some loss in within-laboratory precision for ampoules after Clean-up, particularly for the following isomers: PCB18 (Fig. 6), PCB105 (Fig. 40), PCB114 (Fig. 42), PCB118 (Fig. 44), PCB180 (Fig. 54) and PCB194 (Fig. 56).

As Laboratory 9334B indicated that this was a new procedure when they submitted these results, it is important that they monitor their calibration standards and recovery checks carefully. Changing a laboratory method that "is better" than the previous one, without clearly defining "how much better", may result in changes to a long-term data set such as IADN that are an artifact of the laboratory, not a true change in the environment.

Laboratory 9334C

The method used by Laboratory 9334 reported as "9334C" combines the detection system of the 9334A method and the single fraction Clean-up of the 9334B method, except for the final volume of the sample (Table 5). The same calibration standard was used for all three sets of analyses. For the injection-ready solutions (ampoules 93-3-PCB1 and 93-3-PCB2), 75% of the results were in the range 75-110% of the target (Table 6 and Fig. 59). This is a little better than what was achieved originally for the "9334A" results and there were no "ND" or "0" reported for the isomers on their target list. This suggests that the GC conditions had been modified or that more care was taken with the repeat analysis. However they were biased low relative to the target for PCB4 (Fig. 1) PCB8 (Fig. 3), PCB 77 (Fig. 31) and PCB194 (Fig. 55). Laboratory 9334C was also the only laboratory to report a value for PCB75. This isomer had been on the original proposed list of target isomers (see beginning of Overview section) but was not included in the final preparation of the ampoules. As there were several isomers in the solutions that were not on this laboratory's target list, one of those isomers may elute close to where PCB75 elutes and the peak may have been misidentified.

Within-laboratory precision for the injection-ready solutions was good for all but four isomers, PCB33 (Fig. 11), PCB53 (Fig. 25), PCB66 (Fig. 27), and PCB97 (Fig. 35).

The Clean-up procedure resulted in an upward shift in the results for Laboratory 9334C. While 53% of the results for ampoules 93-3-PCB3 and 93-3-PCB4 are in the

range 75-110% of the target, there are 25% of the results in the range 110-130% of the target and 10% of the results greater than 130% of the target (Table 6 and Fig. 60). Both ampoules were diluted by a factor of 5. The upward shift in the results may be due to some contamination from the solvent used to prepared the dilution. While the results for isomers PCB4 and PCB77 are still low, they may also be considered erratic (Fig. 2 and 32, respectively), as PCB4 and PCB77 was not detected in ampoule 93-3-PCB3. Overall within-laboratory precision deteriorated for the ampoules processed through Clean-up. One pattern of erratic performance was the result for ampoule 93-3-PCB3 being high and the result for ampoule 93-3-PCB4 being close to the target. This pattern is evident for isomers PCB42 (Fig. 16), PCB44 (Fig. 18), PCB47 (Fig. 20), PCB49 (Fig. 22), PCB66 (Fig. 28), PCB70 (Fig. 30), PCB114 (Fig. 42), and PCB136 (Fig. 46). Other isomers such as PCB31 (Fig. 10), PCB40 (Fig. 14), PCB105 (Fig. 40), PCB118 (Fig. 44), PCB153 (Fig. 50), and PCB156 (Fig. 52) show a pattern of the result for ampoule 93-3-PCB3 being at or close to the target but the result for ampoule 93-3-PCB4 being biased low. As the Clean-up procedure was changed to a single fraction analysis, it appears that the method still requires refinement to eliminate some of the variability at different concentration levels. The degree of variable performance demonstrated in this study would not be suitable for the IADN or any other database.

As noted above, better agreement with the target using GC/ECD, for the injection-ready ampoules (93-3-PCB1 and 93-3-PCB2), was achieved with the second set of results (9334C compared to 9334A). Performance using GC/MS (9334B) showed a tendency for a low bias. Laboratory 9334 should carefully establish the comparability of the two instruments before changing their method, as this may have considerable effect on long term databases.

The variability of the results for the ampoules processed through Clean-up (ampoules 93-3-PCB3 and 93-3-PCB4) indicated that further development work is needed to better establish the performance of the different procedures. The original "dry-packed" column used for the "A" results appears to cause losses of the target compounds, though over-dilution may also be a contributing factor. The combination of the single-fraction, "wet-packed", GC/MS analysis appears to produce the more reliable results (set "B"). The single-fraction, "wet-packed", GC/ECD analysis had the most erratic performance, with losses or increases of some target analytes in one ampoule but not the other (set "C"). The single-fraction analysis may result in co-elution of target compounds that were separated in the previous two fractions, causing quantitation problems in the single fraction analysis. The GC/MS detection system may be better able to resolve these co-eluting compounds, though other problems in comparability between GC/ECD and GC/MS may have to be resolved.

Laboratory 9335

Laboratory 9335 had almost 48% of their results for the injection-ready solutions (ampoules 93-3-PCB1 and 93-3-PCB2) in the range 75-110% of the target (Table 6 and Fig. 59). There was a tendency for a high bias for several isomers, as almost 24% of their results were greater than 130% of the target. Isomers that were high were PCB8 (Fig. 3), PCB66 (Fig. 27), PCB70 (Fig. 29), PCB84 (Fig. 33), PCB105 (Fig. 39), and PCB195 (Fig. 57), as well as PCB5, PCB22, PCB37, PCB100, and PCB119, which were not graphed in Youden plots. However, except for PCB22, PCB66, PCB100, and PCB119, the other biased high isomers were all flagged as co-eluting with another isomer. Included with the results was a comment from Laboratory 9335 warning of

possible errors in quantitation for the co-eluting isomers. Isomers that were biased low were PCB4 (Fig. 1) and PCB136 (Fig. 45), as well as PCB18, PCB81, and PCB97 that were not plotted. However PCB4 was the only isomer of these flagged as co-eluting with another compound.

Within laboratory precision was very good. Only two isomers did not have their data points on or close to the diagonal line in the Youden plots, PCB105 (Fig. 39) and PCB153 (Fig. 49).

Laboratory 9335 had almost 44% of their results for the ampoules processed through Clean-up (ampoules 93-3-PCB3 and 93-3-PCB4) in the range 75-110% of the target (Table 6 and Fig. 60). There is an upward shift to the range 110-130% of target compared to the injection-ready solutions, but there is no dramatic shift for any individual isomer. The same group of isomers that were biased high for the injection-ready solutions are biased high for the ampoules processed through Clean-up. However only three of the isomers that were low for the injection-ready solutions are also low for the Clean-up solutions, PCB4 (Fig. 2), PCB136 (Fig. 46) and PCB81 that was not plotted. Within-laboratory precision was maintained for most isomers, except for PCB40 (Fig. 14), PCB105 (Fig. 40), and PCB153 (Fig. 50), and also PCB32 and PCB37 that were not plotted.

Laboratory 9336

Laboratory 9336 had approximately 77% of their results for the injection-ready solutions (ampoules 93-3-PCB1 and 93-3-PCB2) in the range 75-110% of the target (Table 6 and Fig. 59). Isomers that were biased high were PCB4 (Fig. 1) and PCB5, PCB8, and PCB74 that were not graphed in Youden plots. Isomers that were biased low were PCB40 (Fig. 13), PCB47 (Fig. 19), PCB114 (Fig. 41), PCB138 (Fig. 47), and PCB32 and PCB48 that were not plotted. A note was included with the results indicating that for ampoule 93-3-PCB1, the peaks for isomers PCB16 and PCB32 were not resolved, but for ampoule 93-3-PCB2 they were resolved. This would account for the low bias for isomer PCB32 and the somewhat erratic performance for PCB16. Within-laboratory precision was good, with all of the data points in the Youden plots on or close to the diagonal line, except for PCB138 (Fig. 47), which was not as precise. Other isomers not plotted that may also have been less precise were PCB37 and PCB41 (Tables 1 and 2).

Laboratory 9336 maintained their agreement with the target for the ampoules processed through Clean-up (ampoules 93-3-PCB3 and 93-3-PCB4), having 76% of their results in the range 75-110% of the target (Table 6 and Fig. 60). There was a slight shift to the range 60-75% of the target. The same isomers listed above that were biased high or low had similar results in ampoules 93-3-PCB3 and 93-3-PCB4, though agreement with the target improved for isomers PCB5, PCB8, and PCB32. However there appeared to be some losses for isomer PCB22 (Tables 3 and 4). Within-laboratory precision was maintained, except for isomers PCB101 (Fig. 38) and PCB136 (Fig. 46).

5 CONCLUSION

The results from this study demonstrate an interlaboratory mean and median that agree with the target within $\pm 15\%$ for the majority of the PCB isomers in this study, and within $\pm 30\%$ of the target for the remainder. This is similar to the first study⁴ among these participating laboratories. However the between-laboratory variability has not improved from the first study, with most isomers having a %RSD in the range 20-40%. This is larger than is considered an acceptable degree of variability in a similar interlaboratory study⁵. Problems in calibration appeared to be the main source of this variability, particularly for Laboratories 9333 and 9335. Laboratory 9334A also had problems with several "ND" results reported. These results re-emphasize the need for a common reference standard to be used by all of the laboratories contributing to the IADN database so as to avoid biases in long term data sets.

The results from the solutions processed through analytical Clean-up also demonstrated good agreement with the target for the interlaboratory mean and median. However the between-laboratory variability was again greater than 20%. Some individual laboratories demonstrated some problems with their Clean-up procedure, primarily some degree of loss of the target compounds (Laboratory 9332 and 9334A), though it varied from isomer to isomer. Some contamination being added during the Clean-up procedure (Laboratory 9334C), also contributing to the source of error and greater between-laboratory variability.

6 REFERENCES

1. International Joint Commission, United States and Canada; January 1988. *Revised Great Lakes Water Quality Agreement of 1978 as amended by Protocol signed November 18, 1987*.
2. Canada/U.S. Coordinating Committee on Annex 15; March 1990. *Integrated Atmospheric Deposition Network Implementation Plan*.
3. *Integrated Atmospheric Deposition Network Quality Assurance Program Plan (DRAFT)*; May 1993.
4. Cussion, S.; January 1994; *Interlaboratory Study 92-1, Polychlorinated Biphenyl (PCB) Isomers Standard Solutions in Support of the Integrated Atmospheric Deposition Network (IADN), September 1992*; Ontario Ministry of Environment and Energy and Atmospheric Environment Service, Environment Canada. ISBN 0-7778-2242-3.
5. Mes, J., Conacher, H.B.S., and Malcolm, S.; 1993; "An International Study on Feasibility of Estimating Polychlorinated Biphenyls by using Specific Polychlorinated Biphenyl Congeners"; *Inter. J. Environ. Anal. Chem.* 50: 285-297.
6. Youden, W.J. and Steiner, E.H.; 1975. *Statistical Manual of the Association of Official Analytical Chemists*. Association of Official Analytical Chemists; ISBN 0-935584-14-3.

7 APPENDIX 1 - RESULTS AND GRAPHS

Table 1	Ampoule 93-3-PCB1, Results in ng/mL
Table 2	Ampoule 93-3-PCB2, Results in ng/mL
Table 3	Ampoule 93-3-PCB3, Results in ng/mL
Table 4	Ampoule 93-3-PCB4, Results in ng/mL
Table 5	Instruments, GC Columns and Clean-up Conditions
Table 6	Distribution of Participants' Results Relative to Target
Table 7	Percent Recovery of Ampoules Processed Through Analytical Clean-up: 93-3-PCB3
Table 8	Percent Recovery of Ampoules Processed Through Analytical Clean-up: 93-3-PCB4
Figures 1 - 58	Youden Plots
Figures 59 - 60	Distribution of Participants' Results Relative to Target

TABLE 1 - INTERLABORATORY STUDY 93-3
AMPOULE 93-3-PCB1 (ng/mL)

ISOMER	93-3-PCB1	LABORATORY ID CODE							MEAN	MEDIAN	STD DEV	%RSD	n
	TARGET	9332	9333	9334A	9334B	9334C	9335	9336					
PCB4	3.94	3.31	3.7	5.8	2.6	2.1	1.3 §	7.1	3.70	3.31	2.068	55.9%	7
PCB5	3.74	3.35	4.55	N/A	N/A	N/A	9.5 *	4.85 *	5.56	4.7	2.704	48.6%	4
PCB8	3.65	4.09	3.79	5.1	6.4 +	2.8	9.5 *	4.85 *	5.22	4.85	2.200	42.2%	7
PCB16	3.62	2.94	4.685 #	N/A	N/A	N/A	4.75 #	2.6 #	3.74	3.8125	1.133	30.3%	4
PCB17	3.17	2.75	4.41	N/A	N/A	N/A	3.8	2.9	3.47	3.35	0.782	22.6%	4
PCB18	3.33	2.42	3.74	3.3	6.3	3.8	2.5	N/A	3.68	3.3	1.415	38.5%	6
PCB22	4.29	3.09	4.71	N/A	N/A	N/A	6.6	3.2	4.40	3.955	1.642	37.3%	4
PCB28	5.80	4.37	6.90	5.4	4.0	6.0	5.15 &	4.55 &	5.20	5.15	1.010	19.4%	7
PCB31	4.84	3.17	5.66	3.0	3.3	4.2	5.15 &	4.55 &	4.14	4.2	1.034	25.0%	7
PCB32	4.16	3.64	4.685 #	N/A	N/A	N/A	4.75 #	2.6 #	3.92	4.1625	1.016	25.9%	4
PCB33	4.12	3.39	4.28	3.0	3.9	3.7	4.9	3.4	3.80	3.7	0.636	16.8%	7
PCB37	3.80	3.32	N/A	N/A	N/A	N/A	5.75 @	6.5 @	5.19	5.75	1.662	32.0%	3
PCB40	4.32	3.32	5.38	3.6	3.0	4.0	4.6	3.0	3.84	3.6	0.886	23.1%	7
PCB41	4.16	3.45	2.025 %	N/A	N/A	N/A	4.4 %	3.3 %	3.29	3.375	0.976	29.6%	4
PCB42	7.91	6.66	10.43	7.5	5.9	8.3	5.75 @	6.5 @	7.29	6.66	1.646	22.6%	7
PCB44	6.60	5.47	8.30	6.6	5.5	6.8	7.3	5.8	6.54	6.6	1.042	15.9%	7
PCB47	8.22	6.33	8.03	5.4	6.4	8.4	7.25 §	2.7 §	6.36	6.4	1.918	30.2%	7
PCB48	6.06	4.85	6.13	N/A	N/A	N/A	7.25 §	2.7 §	5.23	5.49	1.952	37.3%	4
PCB49	5.94	5.42	8.07	5.0	4.3	5.4	5.8	5.7	5.67	5.42	1.172	20.7%	7
PCB52	5.94	5.02	7.78	6.5	5.6	7.3	5.2	5.8	6.17	5.8	1.057	17.1%	7
PCB53	6.30	3.43	N/A	0.0	5.5	4.7	6.0	6.4	4.34	5.1	2.372	54.7%	6
PCB61	3.39	2.45	N/A	2.6	2.8	3.2	N/A	N/A	2.76	2.7	0.325	11.8%	4
PCB66	4.74	3.82 c	5.25	0.0	3.5	10.3	6.3	4.2	4.77	4.2	3.129	65.6%	7
PCB70	4.32	3.91	5.19	4.8	3.5	4.2	5.8 §	3.8	4.46	4.2	0.834	18.7%	7
PCB71	3.55	3.49	2.025 %	N/A	N/A	N/A	4.4 %	3.3 %	3.30	3.395	0.978	29.6%	4
PCB74	3.36	3.71	2.82	N/A	N/A	N/A	2.9	5.2	3.66	3.305	1.104	30.2%	4
PCB75						3.6							
PCB77	3.39	3.46	N/A	9.3	2.5	1.6	3.7	3.4	3.99	3.43	2.714	68.0%	6
PCB81	3.10	4.17 c	N/A	N/A	N/A	N/A	1.7	2.7	2.86	2.7	1.242	43.5%	3
PCB84	6.24	7.14	N/A	5.6	6.0	6.1	10.8 §	6.2	6.97	6.15	1.942	27.9%	6
PCB95	6.38	6.26	9.79	N/A	N/A	N/A	7.3	6.6	7.49	6.95	1.595	21.3%	4
PCB97	6.14	3.58	8.12	5.9	7.1	6.4	5.0	5.9	6.00	5.9	1.457	24.3%	7
PCB100	5.47	5.07	N/A	N/A	N/A	N/A	7.4	5.4	5.96	5.4	1.261	21.2%	3

TABLE 1 - INTERLABORATORY STUDY 93-3
AMPOULE 93-3-PCB1 (ng/mL)

ISOMER	93-3-PCB1	LABORATORY ID CODE							MEAN	MEDIAN	STD DEV	%RSD	n
	TARGET	9332	9333	9334A	9334B	9334C	9335	9336					
PCB101	5.04	4.54	7.44	5.1	3.8	5.0	4.4	5.0	5.04	5.0	1.152	22.9%	7
PCB105	4.56	3.33	6.99	3.6	3.5	3.9	6.9 ▶	3.8	4.57	3.8	1.630	35.6%	7
PCB110	5.28	2.87 c	7.94	N/A	N/A	N/A	4.4	4.8	5.00	4.6	2.128	42.5%	4
PCB114	4.19	3.83	5.06	3.3	3.4	4.0	4.6 §	3.1	3.90	3.83	0.719	18.4%	7
PCB118	3.23	2.36	4.75	2.7	3.0	2.9	3.3	3.6 ◀	3.23	3.0	0.780	24.2%	7
PCB119	3.97	3.40	N/A	N/A	N/A	N/A	6.3	3.5	4.40	3.5	1.646	37.4%	3
PCB126	3.84	1.58	N/A	N/A	N/A	N/A	N/A	3.8	2.69	-	1.570	58.4%	2
PCB136	6.40	4.72	8.17	5.7	4.9	6.2	3.7	6.6	5.71	5.7	1.458	25.5%	7
PCB138	3.17	1.64	4.56	0.0	3.1	3.0	3.6 §	1.3	2.46	3.0	1.554	63.2%	7
PCB149	3.90	3.33	5.47	N/A	N/A	N/A	3.6	3.6 ◀	4.00	3.6	0.988	24.7%	4
PCB153	7.80	5.88	10.14	6.8	6.4	7.1	6.9 ▶	7.4	7.23	6.9	1.374	19.0%	7
PCB156	5.68	4.56	7.68	4.8	4.8	4.6	N/A	4.5	5.16	4.7	1.242	24.1%	6
PCB169	3.80	2.39	N/A	N/A	N/A	N/A	N/A	N/A	-	-	-	-	1
PCB180	4.68	2.83	6.22	4.3	4.1	3.8	5.1	4.1	4.35	4.1	1.065	24.5%	7
PCB194	4.40	3.87	5.59	4.0	3.3	3.3	4.0	3.6	3.95	3.87	0.782	19.8%	7
PCB195	5.08	4.08	6.73	4.8	4.4	4.3	11.8 §	4.2	5.76	4.4	2.816	48.9%	7

Legend

*, #, \$, %, &, @, ▶, ◀

Reported value was total of co-eluting isomers. Results have been evenly divided among the isomers for the purposes of statistical calculations.

+

Value expected to be high - includes co-eluting compound not on current target list.

c

Peak merged with another identified component; sum of peaks quantified using composite response factor from standard run; value determined by subtraction of value amount for co-eluting peak as determined independently from run on other column. Results prone to large error.

§

Co-elutes with another isomer not on the target list of this interlaboratory study

TABLE 2 - INTERLABORATORY STUDY 93-3
AMPOULE 93-3-PCB2 (ng/mL)

ISOMER	93-3-PCB2	LABORATORY ID CODE							MEAN	MEDIAN	STD DEV	%RSD	n
	TARGET	9332	9333	9334A	9334B	9334C	9335	9336					
PCB4	17.22	14.20	15.09	22.0	9.0	8.4	4.0 §	27.8	14.36	14.20	8.278	57.7%	7
PCB5	16.38	15.00	17.72	N/A	N/A	N/A	38.0 *	20.0 *	22.68	18.86	10.416	45.9%	4
PCB8	15.96	14.84	14.91	21.0	27.2 +	11.0	38.0 *	20.0 *	20.99	20.0	9.160	43.6%	7
PCB16	15.82	12.23	13.595 #	N/A	N/A	N/A	14.6 #	16.9	14.33	14.098	1.969	13.7%	4
PCB17	13.86	12.91	17.37	N/A	N/A	N/A	14.0	11.7	14.00	13.455	2.438	17.4%	4
PCB18	14.56	10.57	15.20	14.0	19.3	16.2	9.5	N/A	14.13	14.6	3.641	25.8%	6
PCB22	18.76	14.09	18.10	N/A	N/A	N/A	28.6	16.2	19.25	17.15	6.447	33.5%	4
PCB28	14.50	10.92	16.27	11.6	10.9	14.5	13.55 &	11.7 &	12.78	11.7	2.053	16.1%	7
PCB31	12.10	8.28	13.26	7.7	8.8	10.3	13.55 &	11.7 &	10.51	10.3	2.382	22.7%	7
PCB32	10.40	9.17	13.595 #	N/A	N/A	N/A	14.6 #	8.8	11.54	11.383	2.984	25.9%	4
PCB33	10.30	8.45	11.38	7.4	8.7	13.0	12.7	9.5	10.16	9.5	2.203	21.7%	7
PCB37	9.50	8.51	N/A	N/A	N/A	N/A	9.1 @	9.3 @	8.97	9.1	0.411	4.6%	3
PCB40	10.80	8.47	13.0	8.2	8.5	9.3	11.8	8.0	9.61	8.5	1.979	20.6%	7
PCB41	10.40	8.57	6.68 %	N/A	N/A	N/A	13.6 %	10.5 %	9.84	9.535	2.954	30.0%	4
PCB42	10.56	8.57	14.72	10.4	8.2	10.8	9.1 @	9.3 @	10.16	9.3	2.217	21.8%	7
PCB44	8.80	6.89	10.62	8.4	6.6	9.5	9.5	8.5	8.57	8.5	1.453	17.0%	7
PCB47	10.96	7.80	10.39	6.6	8.7	11.0	9.8 §	3.55 §	8.26	8.7	2.574	31.2%	7
PCB48	8.08	6.24	7.98	N/A	N/A	N/A	9.8 §	3.55 §	6.89	7.11	2.660	38.6%	4
PCB49	7.92	6.42	10.31	6.2	5.7	7.6	8.0	7.5	7.39	7.5	1.537	20.8%	7
PCB52	7.92	6.62	10.20	8.5	7.3	9.1	6.8	7.7	8.03	7.7	1.305	16.2%	7
PCB53	8.40	4.65	N/A	0.0	7.1	10.8	7.6	8.2	6.39	7.4	3.703	57.9%	6
PCB61	12.72	9.90	N/A	9.3	10.5	10.3	N/A	N/A	10.00	10.1	0.529	5.3%	4
PCB66	17.76	15.93 c	20.78	ND	15.3	14.3	24.3	14.8	15.06	15.3	7.598	50.5%	7
PCB70	16.20	14.80	17.11	18.3	12.7	15.4	20.9 §	14.4	16.23	15.4	2.751	17.0%	7
PCB71	13.32	12.68	6.68 %	N/A	N/A	N/A	13.6 %	10.5 %	10.87	11.59	3.078	28.3%	4
PCB74	12.60	13.67	9.89	N/A	N/A	N/A	10.4	21.5	13.87	12.035	5.358	38.6%	4
PCB75						4.4							
PCB77	12.72	11.61	N/A	32.2	8.4	5.7	13.1	12.0	13.84	11.8	9.401	67.9%	6
PCB81	11.64	9.92 c	N/A	N/A	N/A	N/A	6.0	10.2	8.71	9.92	2.348	27.0%	3
PCB84	18.20	19.03	N/A	13.9	14.9	16.8	28.8 §	16.6	18.34	16.7	5.419	29.6%	6
PCB95	18.62	16.81	19.49	N/A	N/A	N/A	19.1	17.2	18.15	18.15	1.341	7.4%	4
PCB97	17.92	9.77	19.83	15.6	16.0	9.8	13.4	16.3	14.39	15.6	3.667	25.5%	7
PCB100	15.96	13.71	N/A	N/A	N/A	N/A	20.2	14.5	16.14	14.5	3.541	21.9%	3

**TABLE 2 - INTERLABORATORY STUDY 93-3
AMPOULE 93-3-PCB2 (ng/mL)**

ISOMER	93-3-PCB2	LABORATORY ID CODE							MEAN	MEDIAN	STD DEV	%RSD	n
	TARGET	9332	9333	9334A	9334B	9334C	9335	9336					
PCB101	14.70	12.35	18.25	12.6	12.1	13.2	11.7	13.4	13.37	12.6	2.232	16.7%	7
PCB105	13.30	9.02	19.04	10.5	10.3	10.2	16.05 ▶	10.8	12.27	10.5	3.745	30.5%	7
PCB110	15.40	10.87 c	20.87	N/A	N/A	N/A	11.8	13.4	14.24	12.6	4.545	31.9%	4
PCB114	13.10	12.15	14.12	13.8	10.3	13.0	14.4 §	10.7	12.64	13.0	1.645	13.0%	7
PCB118	10.10	7.50	13.58	9.4	8.3	8.7	10.4	10.6 ◀	9.78	9.4	2.008	20.5%	7
PCB119	12.40	10.56	N/A	N/A	N/A	N/A	19.7	11.5	13.92	11.5	5.028	36.1%	3
PCB126	12.00	8.72	N/A	N/A	N/A	N/A	N/A	11.0	9.86	-	1.612	16.4%	2
PCB136	20.00	14.70	22.61	15.8	16.9	19.2	11.3	19.5	17.14	16.9	3.689	21.5%	7
PCB138	9.90	5.12	12.74	0.0	9.8	9.3	10.9 §	8.9	8.11	9.3	4.259	52.5%	7
PCB149	12.20	9.95	15.54	N/A	N/A	N/A	11.4	10.6 ◀	11.87	11	2.516	21.2%	4
PCB153	15.60	11.88	19.04	13.8	13.4	14.2	16.05 ▶	15.9	14.90	14.2	2.329	15.6%	7
PCB156	11.36	8.77	14.69	9.3	10.2	9.6	N/A	10.0	10.43	9.8	2.150	20.6%	6
PCB169	7.60	5.05	N/A	N/A	N/A	N/A	N/A	N/A	-	-	-	-	1
PCB180	9.36	5.99	11.87	10.4	8.6	7.8	10.5	8.3	9.07	8.6	1.982	21.9%	7
PCB194	8.80	7.95	10.77	8.6	5.9	7.1	8.4	7.8	8.07	8.0	1.495	18.5%	7
PCB195	10.16	7.91	12.89	10.3	9.3	9.2	24.6 §	9.2	11.91	9.3	5.804	48.7%	7

Legend

*, #, \$, %, &, @, ▶, ◀

Reported value was total of co-eluting isomers. Results have been evenly divided among the isomers for the purposes of statistical calculations.

+

Value expected to be high - includes co-eluting compound not on current target list.

c

Peak merged with another identified component; sum of peaks quantified using composite response factor from standard run; value determined by subtraction of value amount for co-eluting peak as determined independently from run on other column. Results prone to large error.

§

Co-elutes with another isomer not on the target list of this interlaboratory study

TABLE 3 - INTERLABORATORY STUDY 93-3
AMPOULE 93-3-PCB3 (ng/mL)

ISOMER	93-3-PCB3	LABORATORY ID CODE							MEAN	MEDIAN	STD DEV	%RSD	n
	TARGET	9332	9333	9334A	9334B	9334C	9335	9336					
PCB4	3.94	3.06	24.45 *	0.0	2.8	ND	1.4 §	6.5	2.29	2.10	2.444	106.6%	6
PCB5	3.74	2.82	0.1	N/A	N/A	N/A	9.15 *	4.4 *	4.12	3.61	3.796	92.2%	4
PCB8	3.65	3.31	1.26	5.2	7.5 +	2.3	9.15 *	4.4 *	4.73	4.4	2.814	59.5%	7
PCB16	3.62	2.38	1.265 #	N/A	N/A	N/A	4.7 #	2.95 #	2.82	2.665	1.433	50.8%	4
PCB17	3.17	2.39	1.89	N/A	N/A	N/A	3.5	2.6	2.60	2.495	0.673	25.9%	4
PCB18	3.33	2.36	1.70	3.6	5.7	4.8	2.4	N/A	3.43	3.0	1.566	45.7%	6
PCB22	4.29	2.72	0.96	N/A	N/A	N/A	6.4	3.0	3.27	2.86	2.274	69.5%	4
PCB28	5.80	3.48	3.40	ND	4.7	6.9	5.05 &	4.15 &	4.64	4.7	1.185	25.5%	7
PCB31	4.84	3.12	3.38	0.0	3.8	4.8	5.05 &	4.15 &	3.47	3.8	1.684	48.5%	7
PCB32	4.16	3.09	1.265 #	N/A	N/A	N/A	4.7 #	2.95 #	3.00	3.02	1.404	46.8%	4
PCB33	4.12	2.87	1.59	2.8	3.6	4.6	4.7	3.4	3.37	3.4	1.086	32.3%	7
PCB37	3.80	2.72	N/A	N/A	N/A	N/A	5.75 @	5.5 @	4.66	5.5	1.682	36.1%	3
PCB40	4.32	3.62	0.33	ND	4.2	4.6	4.7	2.8	2.89	3.6	1.973	68.2%	7
PCB41	4.16	2.87	0.80 %	N/A	N/A	N/A	4.45 %	3.05 %	2.79	2.96	1.504	53.9%	4
PCB42	7.91	5.31	3.75	8.3	7.6	10.4	5.75 @	5.5 @	6.66	5.8	2.237	33.6%	7
PCB44	6.60	4.62	3.45	7.8	5.0	9.2	7.6	5.6	6.18	5.6	2.057	33.3%	7
PCB47	8.22	4.98	4.28	6.2	7.1	10.4	7.2 §	2.55 §	6.10	6.2	2.513	41.2%	7
PCB48	6.06	4.04	3.16	N/A	N/A	N/A	7.2 §	2.55 §	4.24	3.6	2.068	48.8%	4
PCB49	5.94	4.35	4.50	5.4	5.0	7.0	5.7	5.6	5.36	5.4	0.890	16.6%	7
PCB52	5.94	4.63	4.03	7.6	6.2	8.0	5.1	5.9	5.92	5.9	1.480	25.0%	7
PCB53	6.30	2.96	N/A	ND	5.4	13.8	5.9	6.6	5.78	5.7	4.615	79.9%	6
PCB61	3.39	2.08	N/A	2.7	3.5	3.6	N/A	N/A	2.97	3.1	0.717	24.1%	4
PCB66	4.74	3.20 c	3.01	ND	4.0	12.0	5.8	3.9	4.56	3.9	3.712	81.4%	7
PCB70	4.32	3.11	2.87	4.6	4.2	5.4	5.8 §	3.8	4.25	4.2	1.100	25.8%	7
PCB71	3.55	2.88	0.80 %	N/A	N/A	N/A	4.45 %	3.05 %	2.80	2.965	1.505	53.8%	4
PCB74	3.36	3.05	1.62	N/A	N/A	N/A	2.9	5.0	3.14	2.975	1.395	44.4%	4
PCB75						4.4							
PCB77	3.39	2.68	N/A	ND	3.9	ND	4.0	3.1	2.28	2.9	1.834	80.4%	6
PCB81	3.10	2.36 c	N/A	N/A	N/A	N/A	1.8	2.6	2.25	2.36	0.411	18.2%	3
PCB84	6.24	5.66	N/A	6.6	7.2	7.2	10.9 §	5.9	7.24	6.9	1.903	26.3%	6
PCB95	6.38	5.21	7.57	N/A	N/A	N/A	7.3	5.0	6.27	6.255	1.352	21.6%	4
PCB97	6.14	2.95	4.60	6.8	7.1	7.4	5.1	5.7	5.66	5.7	1.591	28.1%	7
PCB100	5.47	4.23	N/A	N/A	N/A	N/A	7.4	5.0	5.54	5.0	1.653	29.8%	3

**TABLE 3 - INTERLABORATORY STUDY 93-3
AMPOULE 93-3-PCB3 (ng/mL)**

ISOMER	93-3-PCB3	LABORATORY ID CODE							MEAN	MEDIAN	STD DEV	%RSD	n
	TARGET	9332	9333	9334A	9334B	9334C	9335	9336					
PCB101	5.04	3.82	4.45	5.7	5.2	6.1	4.4	5.3	5.00	5.2	0.805	16.1%	7
PCB105	4.56	2.64	2.74	ND	4.8	4.5	7.0 +	3.6	3.61	3.6	2.177	60.3%	7
PCB110	5.28	2.92 c	2.59	N/A	N/A	N/A	4.6	4.7	3.70	3.76	1.103	29.8%	4
PCB114	4.19	3.15	2.97	3.4	3.6	4.8	4.6 §	2.9	3.63	3.4	0.771	21.2%	7
PCB118	3.23	1.88	2.95	ND	4.0	3.2	3.5	3.4 ◀	2.70	3.2	1.360	50.3%	7
PCB119	3.97	2.75	N/A	N/A	N/A	N/A	6.4	3.5	4.22	3.5	1.928	45.7%	3
PCB126	3.84	2.04	N/A	N/A	N/A	N/A	N/A	3.3	2.67	-	0.891	33.4%	2
PCB136	6.40	3.91	4.78	7.2	6.0	7.5	3.8	6.7	5.70	6.0	1.541	27.0%	7
PCB138	3.17	1.38	3.05	ND	3.1	3.5	3.9 §	1.4	2.33	3.1	1.423	61.0%	7
PCB149	3.90	2.75	3.58	N/A	N/A	N/A	3.6	3.4 ◀	3.33	3.49	0.399	12.0%	4
PCB153	7.80	4.77	6.80	7.3	7.0	7.8	7.0 +	7.1	6.82	7.0	0.961	14.1%	7
PCB156	5.68	3.56	5.15	ND	6.0	5.4	N/A	4.3	4.07	4.7	2.170	53.3%	6
PCB169	3.80	2.01	N/A	N/A	N/A	N/A	N/A	N/A	-	-	-	-	1
PCB180	4.68	2.53	4.63	4.2	4.2	4.3	5.2	4.0	4.15	4.2	0.817	19.7%	7
PCB194	4.40	3.35	3.46	3.6	4.5	3.8	4.4	3.5	3.80	3.6	0.465	12.2%	7
PCB195	5.08	3.26	5.07	4.8	5.8	5.0	12.6 §	3.9	5.78	5.0	3.122	54.0%	7

Legend

*, #, \$, %, &, @, +, ◀

Reported value was total of co-eluting isomers. Results have been evenly divided among the isomers for the purposes of statistical calculations.

+

Value expected to be high - includes co-eluting compound not on current target list.

c

Peak merged with another identified component; sum of peaks quantified using composite response factor from standard run; value determined by subtraction of value amount for co-eluting peak as determined independently from run on other column. Results prone to large error.

§

Co-elutes with another isomer not on the target list of this interlaboratory study

⊗

Value excluded from statistical calculations.

**TABLE 4 - INTERLABORATORY STUDY 93-3
AMPOULE 93-3-PCB4 (ng/mL)**

ISOMER	93-3-PCB4	LABORATORY ID CODE							MEAN	MEDIAN	STD DEV	%RSD	n
	TARGET	9332	9333	9334A	9334B	9334C	9335	9336					
PCB4	17.22	12.67	14.85	ND	9.7	8.4	4.2 \$	24.2	10.57	9.7	7.813	73.9%	7
PCB5	16.38	13.33	3.25	N/A	N/A	N/A	37.1 *	16.9 *	17.65	15.115	14.200	80.5%	4
PCB8	15.96	13.98	6.52	19.6	28.7 +	11.0	37.1 *	16.9 *	19.11	16.9	10.578	55.3%	7
PCB16	15.82	10.99	4.48 #	N/A	N/A	N/A	14.55 #	15.6	11.41	12.77	5.021	44.0%	4
PCB17	13.86	11.25	7.7	N/A	N/A	N/A	13.4	10.4	10.69	10.825	2.358	22.1%	4
PCB18	14.56	10.16	7.21	18.0	14.8	16.2	9.3	N/A	12.61	12.5	4.309	34.2%	6
PCB22	18.76	13.23	6.72	N/A	N/A	N/A	28.0	14.5	15.61	13.865	8.934	57.2%	4
PCB28	14.50	9.81	8.42	12.4	11.6	12.8	13.0 &	9.9 &	11.13	11.6	1.766	15.9%	7
PCB31	12.10	7.95	7.24	8.6	9.7	10.3	13.0 &	9.9 &	9.53	9.7	1.889	19.8%	7
PCB32	10.40	8.58	4.48 #	N/A	N/A	N/A	14.55 #	8.5	9.03	8.54	4.150	46.0%	4
PCB33	10.30	7.72	5.47	8.0	8.8	13.0	12.3	8.3	9.08	8.3	2.661	29.3%	7
PCB37	9.50	8.15	N/A	N/A	N/A	N/A	9.15 @	9.45 @	8.92	9.15	0.681	7.6%	3
PCB40	10.80	7.62	1.64	ND	9.2	9.3	8.8	7.3	6.27	7.6	3.825	61.1%	7
PCB41	10.40	7.50	3.325 %	N/A	N/A	N/A	12.4 %	9.9 %	8.28	8.7	3.863	46.6%	4
PCB42	10.56	7.68	6.09	11.5	8.5	10.8	9.15 @	9.45 @	9.02	9.2	1.834	20.3%	7
PCB44	8.80	6.34	5.28	12.9	6.8	9.5	9.8	8.0	8.37	8.0	2.586	30.9%	7
PCB47	10.96	6.84	5.69	8.4	9.0	11.0	9.65 \$	3.4 \$	7.71	8.4	2.587	33.5%	7
PCB48	8.08	5.48	4.33	N/A	N/A	N/A	9.65 \$	3.4 \$	5.72	4.905	2.758	48.3%	4
PCB49	7.92	5.91	5.60	7.3	6.3	7.6	7.8	7.2	6.82	7.2	0.869	12.7%	7
PCB52	7.92	6.61	5.57	10.6	7.9	9.1	6.7	7.7	7.74	7.7	1.689	21.8%	7
PCB53	8.40	4.13	N/A	ND	6.9	10.8	7.5	8.0	6.22	7.2	3.723	59.8%	6
PCB61	12.72	8.71	N/A	9.8	12.2	10.3	N/A	N/A	10.25	10.05	1.458	14.2%	4
PCB66	17.76	14.71 c	11.90	ND	17.1	14.3	23.3	13.8	13.59	14.3	7.023	51.7%	7
PCB70	16.20	13.16	10.26	21.4	15.0	15.4	21.2 \$	14.1	15.79	15.0	4.120	26.1%	7
PCB71	13.32	11.53	3.325 %	N/A	N/A	N/A	12.4 %	9.9 %	9.29	10.715	4.109	44.2%	4
PCB74	12.60	12.39	5.81	N/A	N/A	N/A	10.6	20.9	12.43	11.495	6.296	50.7%	4
PCB75						4.4							
PCB77	12.72	10.97	N/A	ND	11.2	5.7	14.4	10.0	8.71	10.5	5.104	58.6%	6
PCB81	11.64	8.79	N/A	N/A	N/A	N/A	6.5	9.8	8.36	8.79	1.691	20.2%	3
PCB84	18.20	16.94	N/A	17.7	17.3	16.8	29.2 \$	16.0	18.99	17.1	5.034	26.5%	6
PCB95	18.62	14.98	12.20	N/A	N/A	N/A	19.2	16.2	15.65	15.59	2.902	18.5%	4
PCB97	17.92	8.74	12.28	19.6	17.7	9.8	13.5	15.7	13.90	13.5	4.008	28.8%	7
PCB100	15.96	12.46	N/A	N/A	N/A	N/A	20.2	13.6	15.42	13.6	4.179	27.1%	3

**TABLE 4 - INTERLABORATORY STUDY 93-3
AMPOULE 93-3-PCB4 (ng/mL)**

ISOMER	93-3-PCB4	LABORATORY ID CODE							MEAN	MEDIAN	STD DEV	%RSD	n
	TARGET	9332	9333	9334A	9334B	9334C	9335	9336					
PCB101	14.70	10.74	11.70	15.9	13.5	13.2	11.9	12.0	12.71	12.0	1.688	13.3%	7
PCB105	13.30	8.02	8.58	9.3	11.2	10.2	15.7 ▶	11.7	10.67	10.2	2.586	24.2%	7
PCB110	15.40	9.98 c	7.82	N/A	N/A	N/A	12.2	12.0	10.50	10.99	2.049	19.5%	4
PCB114	13.10	10.52	9.09	13.8	13.1	13.0	14.1 §	10.7 ◀	12.04	13.0	1.923	16.0%	7
PCB118	10.10	6.75	8.85	8.6	9.6	8.7	10.2	10.1	8.97	8.9	1.179	13.1%	7
PCB119	12.40	9.47	N/A	N/A	N/A	N/A	20.1	10.7	13.42	10.7	5.815	43.3%	3
PCB126	12.00	7.35	N/A	N/A	N/A	N/A	N/A	10.0	8.68	-	1.874	21.6%	2
PCB136	20.00	13.28	14.39	22.4	17.9	19.2	11.4	17.9	16.64	17.9	3.802	22.8%	7
PCB138	9.90	4.60	8.92	ND	9.6	9.2	11.0 §	8.3	7.37	8.9	3.803	51.6%	7
PCB149	12.20	8.62	10.41	N/A	N/A	N/A	11.0	10.1 ◀	10.03	10.255	1.013	10.1%	4
PCB153	15.60	10.56	13.17	17.0	14.8	14.2	15.7 ▶	14.2	14.23	14.2	2.031	14.3%	7
PCB156	11.36	7.72	10.32	8.6	10.8	9.6	N/A	9.7	9.46	9.7	1.130	11.9%	6
PCB169	7.60	4.88	N/A	N/A	N/A	N/A	N/A	N/A	-	-	-	-	1
PCB180	9.36	5.32	8.69	12.0	9.9	7.8	10.8	8.1	8.94	8.7	2.195	24.5%	7
PCB194	8.80	7.36	8.26	9.9	7.3	7.1	9.2	7.4	8.07	7.4	1.092	13.5%	7
PCB195	10.16	7.21	9.68	12.1	12.8	9.2	26.7 §	9.2	12.41	9.7	6.577	53.0%	7

Legend

*, #, \$, %, &, @, ▶, ◀

Reported value was total of co-eluting isomers. Results have been evenly divided among the isomers for the purposes of statistical calculations.

+

Value expected to be high - includes co-eluting compound not on current target list.

c

Peak merged with another identified component; sum of peaks quantified using composite response factor from standard run; value determined by subtraction of value amount for co-eluting peak as determined independently from run on other column. Results prone to large error.

§

Co-elutes with another isomer not on the target list of this interlaboratory study

TABLE 5 - INSTRUMENTS, GC COLUMNS and CLEAN-UP CONDITIONS

Laboratory	Instrument & Detector	GC Column	Clean-up Conditions			
			Sample Volume	Solvents Used	Column Packing Material	Final Sample Volume
9332	HP 5890, Series II with Dual ECD	DB-5: 60 m, 0.25 mm ID, 0.25 μ m (Main) DB-17: 30 m, 0.25 mm ID, 0.25 μ m (Confirmatory)	1.0 cm ³ (PCB3) 0.5 cm ³ (PCB4)	Fraction 1: Hexane Fraction 2: 15% DCM/ 85% Hexane Fraction 3a: 40% DCM/ 60% Hexane Fraction 3b: DCM	Florisil, 80-100 mesh, deactivated with 3% water	Reduced to 0.5 mL; adjusted to 1.0 mL with iso-octane
9333	Varian 3400 with ECD	RTX-5: 60 m, 0.25 mm ID, 0.25 μ m film	5 mL	Hexane	1.2% Deactivated Florisil	5 mL
9334A	HP 5890 with Dual ECD	DB-5: 60 m, 0.25 mm ID, 0.25 μ m DB-1701: 60 m, 0.25 mm ID, 0.25 μ m	1.0 mL	Fraction 1: Hexane Fraction 2: Dichloromethane	Florisil, 100/200 mesh, "dry packed"	5.0 mL
9334B	HP 5890 with HP 5971A MSD	HP-1: 50 m, 0.2 mm ID, 0.33 μ m film	1.0 mL	Dichloromethane	Florisil, 100/200 mesh, "wet packed"	1.0 mL
9334C	HP 5890 with Dual ECD; single fraction analysis	DB-5: 60 m, 0.25 mm ID, 0.25 μ m DB-1701: 60 m, 0.25 mm ID, 0.25 μ m	1.0 mL	Dichloromethane (45 mL)	Florisil, 100/200 mesh, "wet packed in Hexane"	5.0 mL
9335	HP 5890 with ECD, 7673 autosampler	DB-5: 30 m, 0.25 mm ID, 0.25 μ m	1 mL	Fraction 1: Hexane Fraction 2: 40% Dichloromethane in Hexane	Silica Gel, 100-200 mesh, 3% deactivated	1 mL
9336	GC with Dual ECD	DB-1: 30 m DB-5: 30 m	1 mL	Fraction 1: Hexane Fraction 2: 50:50 Dichloromethane & Hexane	Deactivated Florisil	1 mL

TABLE 6 - DISTRIBUTION OF PARTICIPANTS'S RESULTS RELATIVE TO TARGET

	Injection-Ready Solutions													
Lab ID Code	9332		9333		9334A		9334B		9334C		9335		9336	
Target Range	n	%	n	%	n	%	n	%	n	%	n	%	n	%
< 60%	8	8.3%	3	3.9%	3	5.3%	1	1.7%	5	8.3%	6	6.8%	5	5.6%
60-75%	19	19.8%	1	1.3%	7	12.3%	14	23.3%	3	5.0%	3	3.4%	6	6.7%
75-90%	46	47.9%	4	5.3%	20	35.1%	31	51.7%	18	30.0%	15	17.0%	31	34.4%
90-110%	19	19.8%	16	21.1%	18	31.6%	9	15.0%	27	45.0%	27	30.7%	38	42.2%
110-130%	3	3.1%	31	40.8%	4	7.0%	1	1.7%	6	10.0%	16	18.2%	4	4.4%
> 130%	1	1.0%	21	27.6%	5	8.8%	4	6.7%	1	1.7%	21	23.9%	6	6.7%
	Ampoules Processed through Clean-up													
Lab ID Code	9332		9333		9334A		9334B		9334C		9335		9336	
Target Range	n	%	n	%	n	%	n	%	n	%	n	%	n	%
< 60%	14	14.6%	37	48.7%	16	26.7%	1	1.7%	5	8.3%	6	6.8%	5	5.6%
60-75%	48	50.0%	17	22.4%	3	5.0%	1	1.7%	2	3.3%	3	3.4%	10	11.1%
75-90%	29	30.2%	9	11.8%	12	20.0%	24	40.0%	12	20.0%	18	20.5%	45	50.0%
90-110%	5	5.2%	11	14.5%	14	23.3%	25	41.7%	20	33.3%	21	23.9%	23	25.6%
110-130%	0	0.0%	1	1.3%	11	18.3%	6	10.0%	15	25.0%	21	23.9%	2	2.2%
> 130%	0	0.0%	1	1.3%	4	6.7%	3	5.0%	6	10.0%	19	21.6%	5	5.6%

**TABLE 7 - PERCENT RECOVERY OF AMPOULES PROCESSED THROUGH
ANALYTICAL CLEAN-UP: 93-3-PCB3**

	Target (ng/mL)	Laboratory ID Code						
Isomer	93-3-PCB3	9332	9333	9334A	9334B	9334C	9335	9336
PCB4	3.94	77.7%	621.2%	0.0%	71.1%	0.0%	35.6%	165.1%
PCB5	3.74	75.3%	2.7%	N/A	N/A	N/A	244.4%	117.5%
PCB8	3.65	90.7%	34.5%	142.5%	205.6%	63.0%	250.8%	120.6%
PCB16	3.62	65.8%	35.0%	N/A	N/A	N/A	130.0%	81.6%
PCB17	3.17	75.4%	59.7%	N/A	N/A	N/A	110.5%	82.1%
PCB18	3.33	70.9%	51.1%	108.2%	171.3%	144.2%	72.1%	N/A
PCB22	4.29	63.4%	22.4%	N/A	N/A	N/A	149.3%	70.0%
PCB28	5.80	60.0%	58.6%	82.8%	81.0%	119.0%	87.1%	71.6%
PCB31	4.84	64.5%	69.8%	0.0%	78.5%	99.2%	104.3%	85.7%
PCB32	4.16	74.3%	30.4%	N/A	N/A	N/A	113.0%	70.9%
PCB33	4.12	69.7%	38.6%	68.0%	87.4%	111.7%	114.1%	82.5%
PCB37	3.80	71.6%	N/A	N/A	N/A	N/A	151.3%	144.7%
PCB40	4.32	83.8%	7.6%	0.0%	97.2%	106.5%	108.8%	64.8%
PCB41	4.16	69.0%	19.2%	N/A	N/A	N/A	107.0%	73.3%
PCB42	7.91	67.1%	47.4%	104.9%	96.0%	131.4%	72.7%	69.5%
PCB44	6.60	70.0%	52.3%	118.2%	75.8%	139.4%	115.2%	84.8%
PCB47	8.22	60.6%	52.1%	75.4%	86.4%	126.5%	87.6%	31.0%
PCB48	6.06	66.7%	52.1%	N/A	N/A	N/A	118.8%	42.1%
PCB49	5.94	73.2%	75.8%	90.9%	84.2%	117.8%	96.0%	94.3%
PCB52	5.94	77.9%	67.8%	127.9%	104.4%	134.7%	85.9%	99.3%
PCB53	6.30	47.0%	N/A	0.0%	85.7%	219.0%	93.7%	104.8%
PCB61	3.39	61.3%	N/A	79.6%	103.2%	106.1%	N/A	N/A
PCB66	4.74	67.6%	63.6%	0.0%	84.5%	253.4%	122.5%	82.3%
PCB70	4.32	72.0%	66.4%	106.5%	97.2%	125.0%	134.3%	88.0%
PCB71	3.55	81.1%	22.5%	N/A	N/A	N/A	125.3%	85.9%
PCB74	3.36	90.8%	48.2%	N/A	N/A	N/A	86.3%	148.8%
PCB77	3.39	79.0%	N/A	0.0%	115.0%	0.0%	117.9%	91.4%
PCB81	3.10	76.0%	N/A	N/A	N/A	N/A	58.0%	83.8%
PCB84	6.24	90.7%	N/A	105.8%	115.4%	115.4%	174.7%	94.6%
PCB95	6.38	81.6%	118.6%	N/A	N/A	N/A	114.3%	78.3%
PCB97	6.14	48.0%	74.9%	110.7%	115.6%	120.4%	83.0%	92.8%

**TABLE 7 - PERCENT RECOVERY OF AMPOULES PROCESSED THROUGH
ANALYTICAL CLEAN-UP: 93-3-PCB3**

	Target (ng/mL)	Laboratory ID Code						
Isomer	93-3-PCB3	9332	9333	9334A	9334B	9334C	9335	9336
PCB100	5.47	77.3%	N/A	N/A	N/A	N/A	135.2%	91.4%
PCB101	5.04	75.8%	88.3%	113.1%	103.2%	121.0%	87.3%	105.2%
PCB105	4.56	57.9%	60.1%	0.0%	105.3%	98.7%	153.5%	78.9%
PCB110	5.28	55.3%	49.1%	N/A	N/A	N/A	87.1%	89.0%
PCB114	4.19	75.1%	70.8%	81.1%	85.9%	114.5%	109.7%	69.2%
PCB118	3.23	58.2%	91.3%	0.0%	123.8%	99.0%	108.3%	105.2%
PCB119	3.97	69.3%	N/A	N/A	N/A	N/A	161.3%	88.2%
PCB126	3.84	53.1%	N/A	N/A	N/A	N/A	N/A	85.9%
PCB136	6.40	61.1%	74.7%	112.5%	93.8%	117.2%	59.4%	104.7%
PCB138	3.17	43.6%	96.3%	0.0%	97.9%	110.5%	123.1%	44.2%
PCB149	3.90	70.4%	91.7%	N/A	N/A	N/A	92.2%	87.1%
PCB153	7.80	61.2%	87.2%	93.6%	89.7%	100.0%	89.7%	91.0%
PCB156	5.68	62.7%	90.7%	0.0%	105.6%	95.1%	N/A	75.7%
PCB169	3.80	52.9%	N/A	N/A	N/A	N/A	N/A	N/A
PCB180	4.68	54.1%	98.9%	89.7%	89.7%	91.9%	111.1%	85.5%
PCB194	4.40	76.1%	78.6%	81.8%	102.3%	86.4%	100.0%	79.5%
PCB195	5.08	64.2%	99.8%	94.5%	114.2%	98.4%	248.0%	76.8%

**TABLE 8 - PERCENT RECOVERY OF AMPOULES PROCESSED THROUGH
ANALYTICAL CLEAN-UP: 93-3-PCB4**

	Target (ng/mL)	Laboratory ID Code						
Isomer	93-3-PCB4	9332	9333	9334A	9334B	9334C	9335	9336
PCB4	17.22	73.6%	86.2%	0.0%	56.3%	48.8%	24.4%	140.5%
PCB5	16.38	81.4%	19.8%	N/A	N/A	N/A	226.5%	103.2%
PCB8	15.96	87.6%	40.9%	122.8%	179.8%	68.9%	232.5%	105.9%
PCB16	15.82	69.5%	28.3%	N/A	N/A	N/A	92.0%	98.6%
PCB17	13.86	81.2%	55.6%	N/A	N/A	N/A	96.7%	75.0%
PCB18	14.56	69.8%	49.5%	123.6%	101.6%	111.3%	63.9%	N/A
PCB22	18.76	70.5%	35.8%	N/A	N/A	N/A	149.3%	77.3%
PCB28	14.50	67.7%	58.1%	85.5%	80.0%	88.3%	89.7%	68.3%
PCB31	12.10	65.7%	59.8%	71.1%	80.2%	85.1%	107.4%	81.8%
PCB32	10.40	82.5%	43.1%	N/A	N/A	N/A	139.9%	81.7%
PCB33	10.30	75.0%	53.1%	77.7%	85.4%	126.2%	119.4%	80.6%
PCB37	9.50	85.8%	N/A	N/A	N/A	N/A	96.3%	99.5%
PCB40	10.80	70.6%	15.2%	0.0%	85.2%	86.1%	81.5%	67.6%
PCB41	10.40	72.1%	32.0%	N/A	N/A	N/A	119.2%	95.2%
PCB42	10.56	72.7%	57.7%	108.9%	80.5%	102.3%	86.6%	89.5%
PCB44	8.80	72.0%	60.0%	146.6%	77.3%	108.0%	111.4%	90.9%
PCB47	10.96	62.4%	51.9%	76.6%	82.1%	100.4%	88.0%	31.0%
PCB48	8.08	67.8%	53.6%	N/A	N/A	N/A	119.4%	42.1%
PCB49	7.92	74.6%	70.7%	92.2%	79.5%	96.0%	98.5%	90.9%
PCB52	7.92	83.5%	70.3%	133.8%	99.7%	114.9%	84.6%	97.2%
PCB53	8.40	49.2%	N/A	0.0%	82.1%	128.6%	89.3%	95.2%
PCB61	12.72	68.5%	N/A	77.0%	95.9%	81.0%	N/A	N/A
PCB66	17.76	82.8%	67.0%	0.0%	96.3%	80.5%	131.2%	77.7%
PCB70	16.20	81.2%	63.3%	132.1%	92.6%	95.1%	130.9%	87.0%
PCB71	13.32	86.6%	25.0%	N/A	N/A	N/A	93.1%	74.3%
PCB74	12.60	98.3%	46.1%	N/A	N/A	N/A	84.1%	165.9%
PCB77	12.72	86.2%	N/A	0.0%	88.1%	44.8%	113.2%	78.6%
PCB81	11.64	75.5%	N/A	N/A	N/A	N/A	55.8%	84.2%
PCB84	18.20	93.1%	N/A	97.3%	95.1%	92.3%	160.4%	87.9%
PCB95	18.62	80.5%	65.5%	N/A	N/A	N/A	103.1%	87.0%
PCB97	17.92	48.8%	68.5%	109.4%	98.8%	54.7%	75.3%	87.6%

**TABLE 8 - PERCENT RECOVERY OF AMPOULES PROCESSED THROUGH
ANALYTICAL CLEAN-UP: 93-3-PCB4**

	Target (ng/mL)	Laboratory ID Code						
Isomer	93-3-PCB4	9332	9333	9334A	9334B	9334C	9335	9336
PCB100	15.96	78.1%	N/A	N/A	N/A	N/A	126.6%	85.2%
PCB101	14.70	73.1%	79.6%	108.2%	91.8%	89.8%	81.0%	81.6%
PCB105	13.30	60.3%	64.5%	69.9%	84.2%	76.7%	118.0%	88.0%
PCB110	15.40	64.8%	50.8%	N/A	N/A	N/A	79.2%	77.9%
PCB114	13.10	80.3%	69.4%	105.3%	100.0%	99.2%	107.6%	81.7%
PCB118	10.10	66.8%	87.6%	85.1%	95.0%	86.1%	101.0%	100.0%
PCB119	12.40	76.4%	N/A	N/A	N/A	N/A	162.1%	86.3%
PCB126	12.00	61.3%	N/A	N/A	N/A	N/A	N/A	83.3%
PCB136	20.00	66.4%	72.0%	112.0%	89.5%	96.0%	57.0%	89.5%
PCB138	9.90	46.5%	90.1%	0.0%	97.0%	92.9%	111.1%	83.8%
PCB149	12.20	70.7%	85.3%	N/A	N/A	N/A	90.2%	82.8%
PCB153	15.60	67.7%	84.4%	109.0%	94.9%	91.0%	100.6%	91.0%
PCB156	11.36	68.0%	90.8%	75.7%	95.1%	84.5%	N/A	85.4%
PCB169	7.60	64.2%	N/A	N/A	N/A	N/A	N/A	N/A
PCB180	9.36	56.8%	92.8%	128.2%	105.8%	83.3%	115.4%	86.5%
PCB194	8.80	83.6%	93.9%	112.5%	83.0%	80.7%	104.5%	84.1%
PCB195	10.16	71.0%	95.3%	119.1%	126.0%	90.6%	262.8%	90.6%

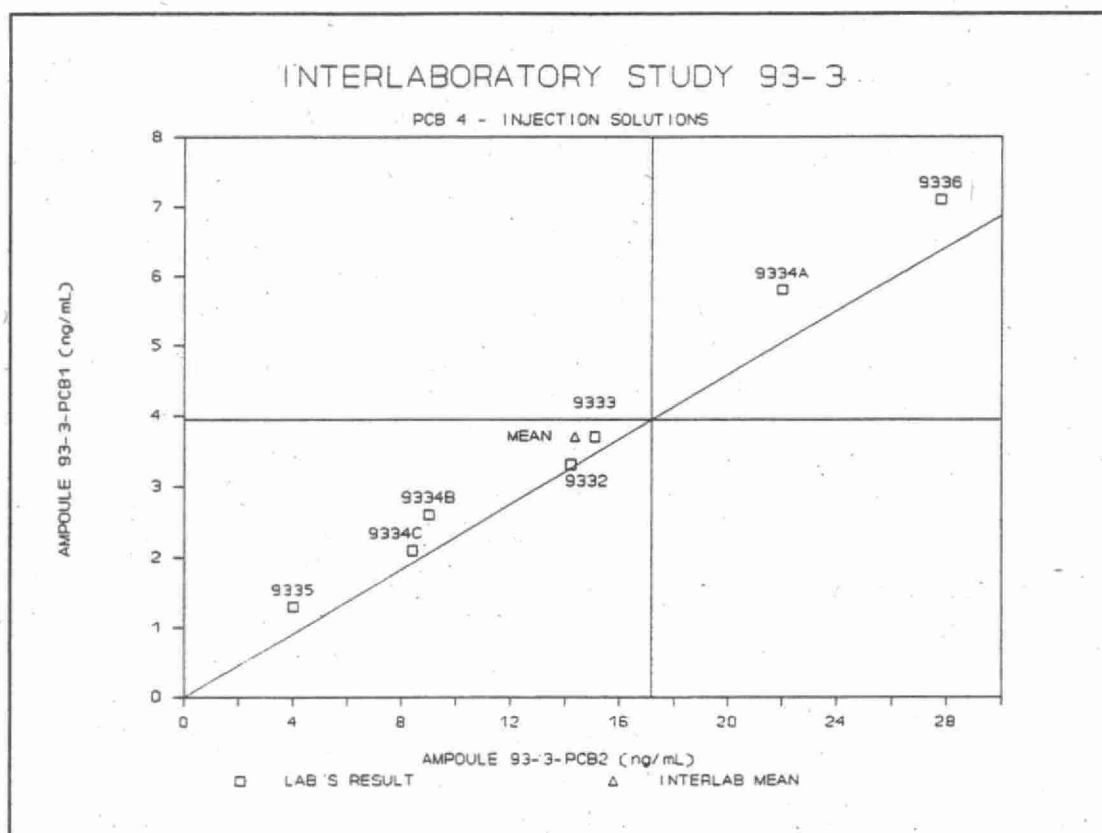


Figure 1 - PCB4 from Injection-Ready Ampoules

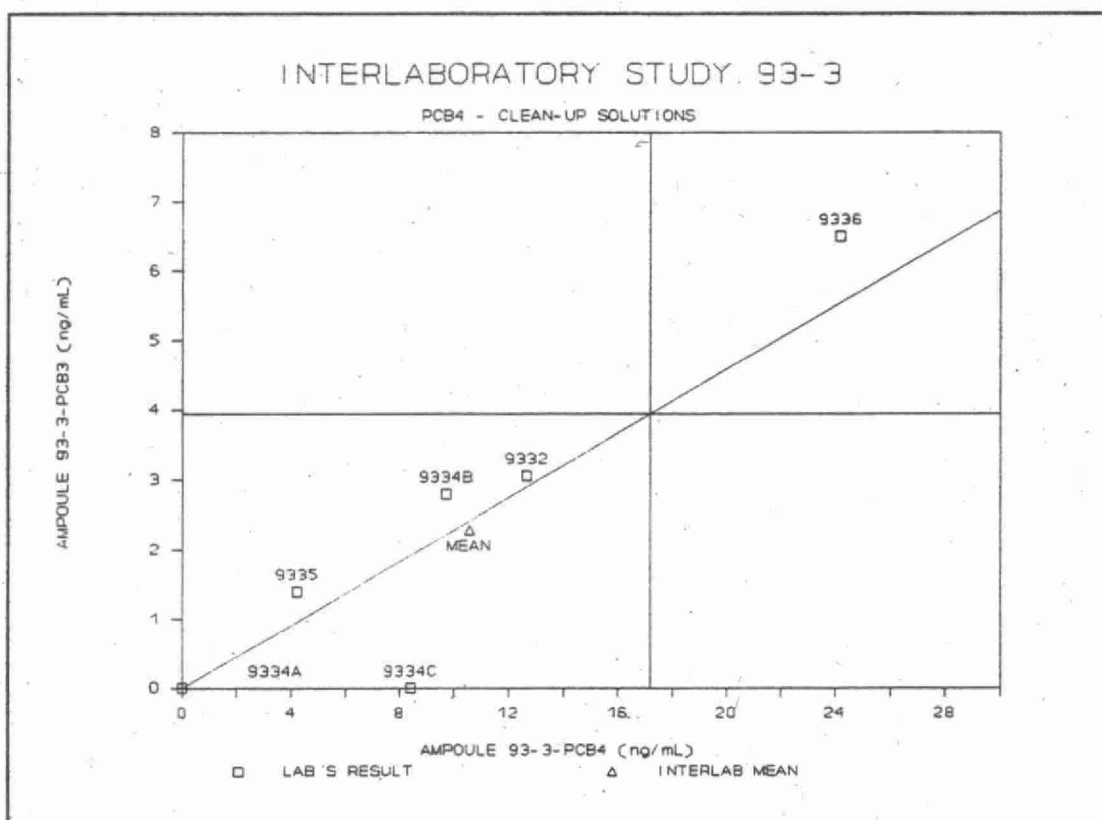


Figure 2 - PCB4 from Ampoules Processed through Clean-up

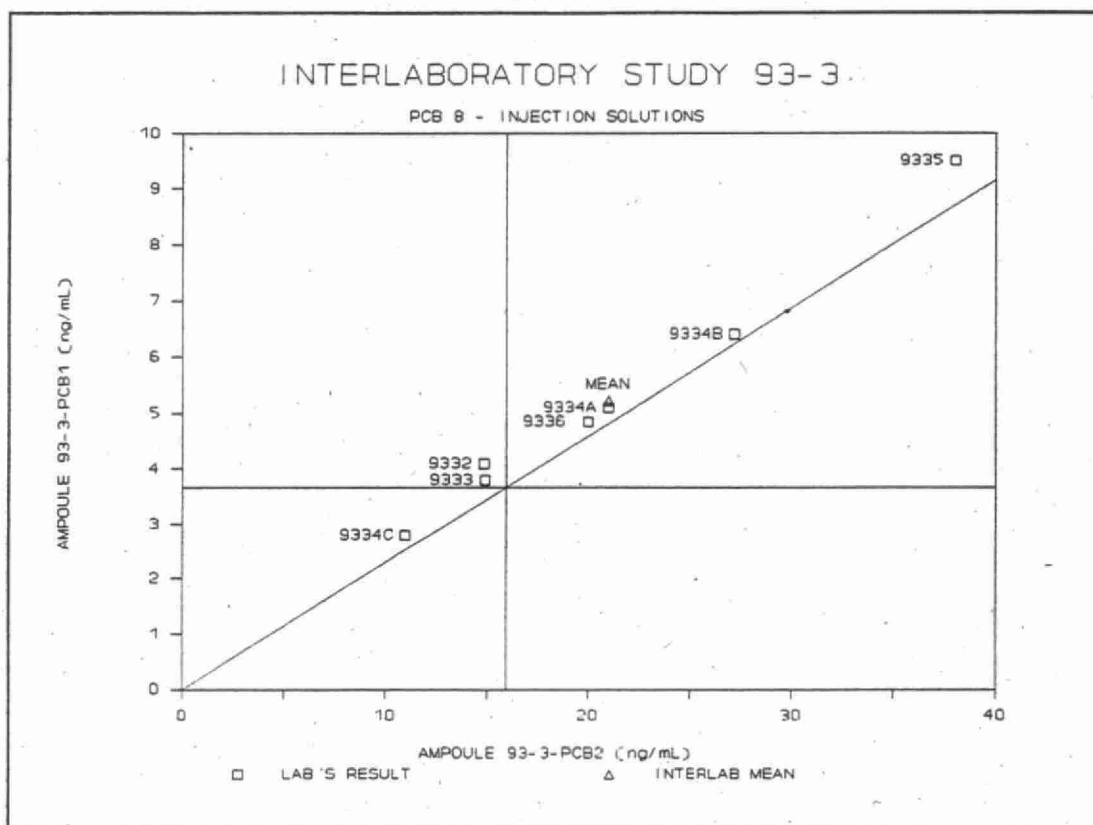


Figure 3 - PCB8 from Injection-Ready Ampoules

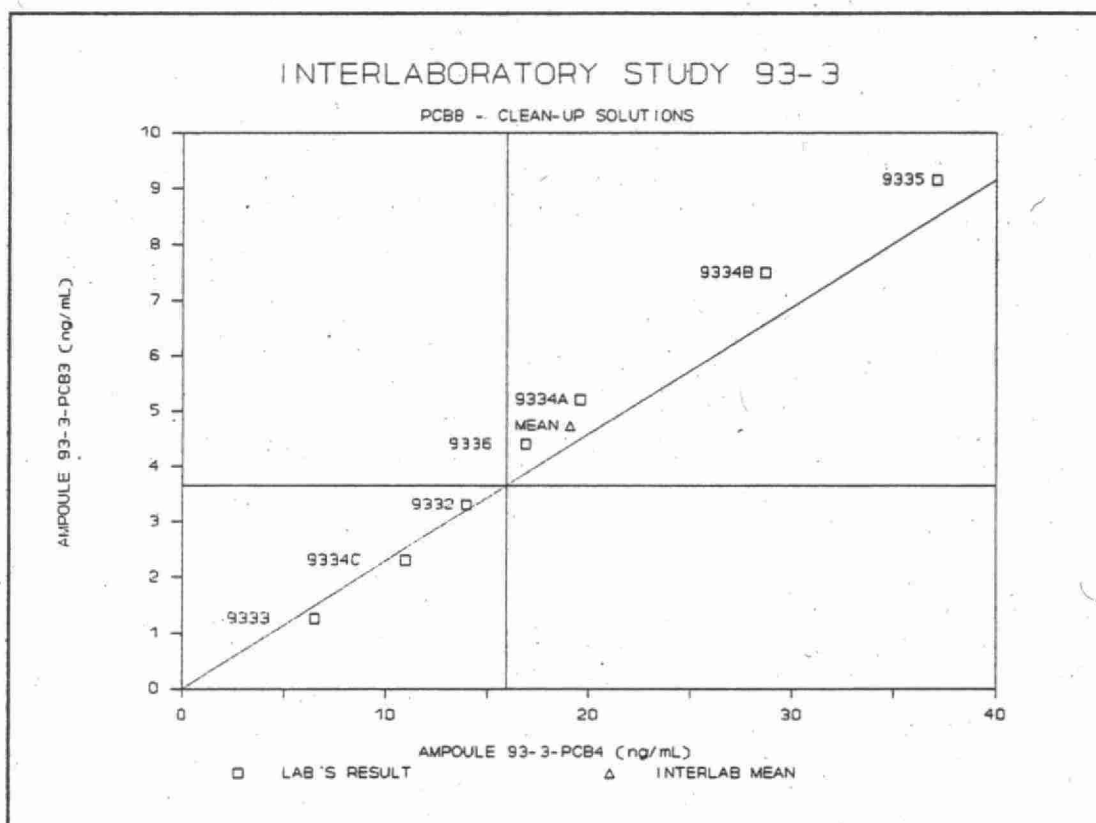


Figure 4 - PCB8 from Ampoules Processed Through Clean-up

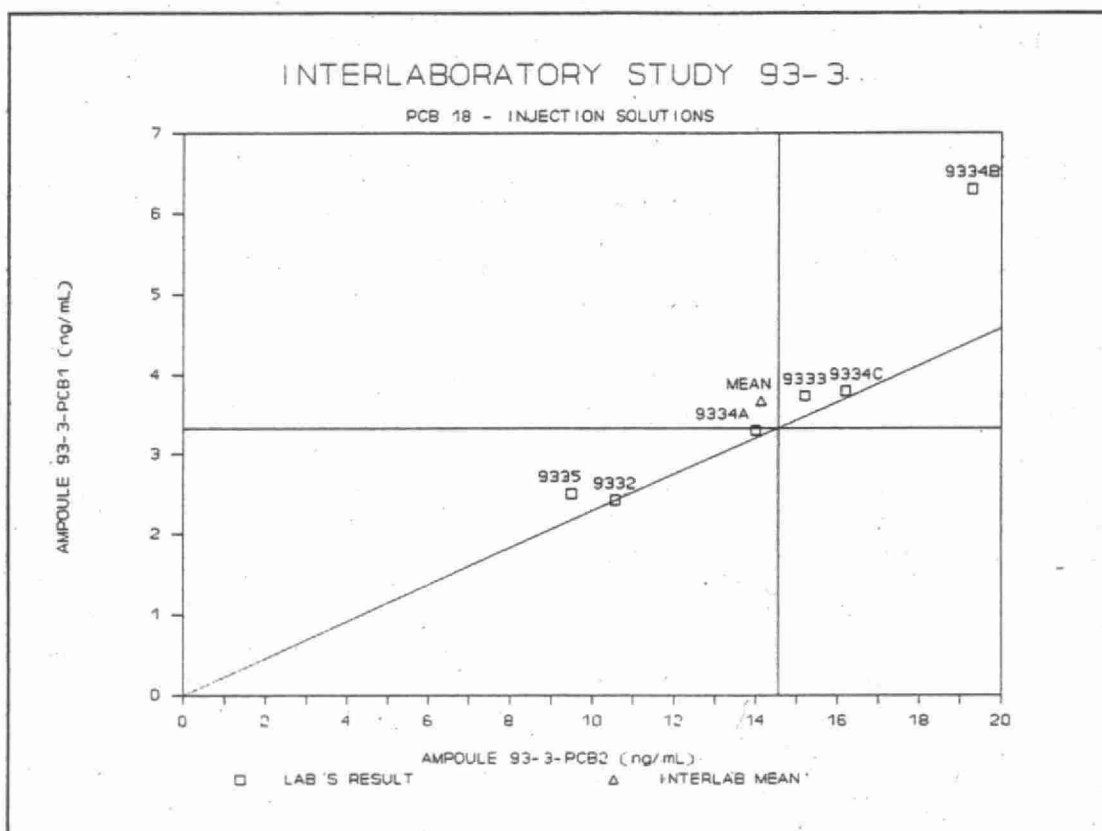


Figure 5 - PCB18 from Injection-Ready Ampoules

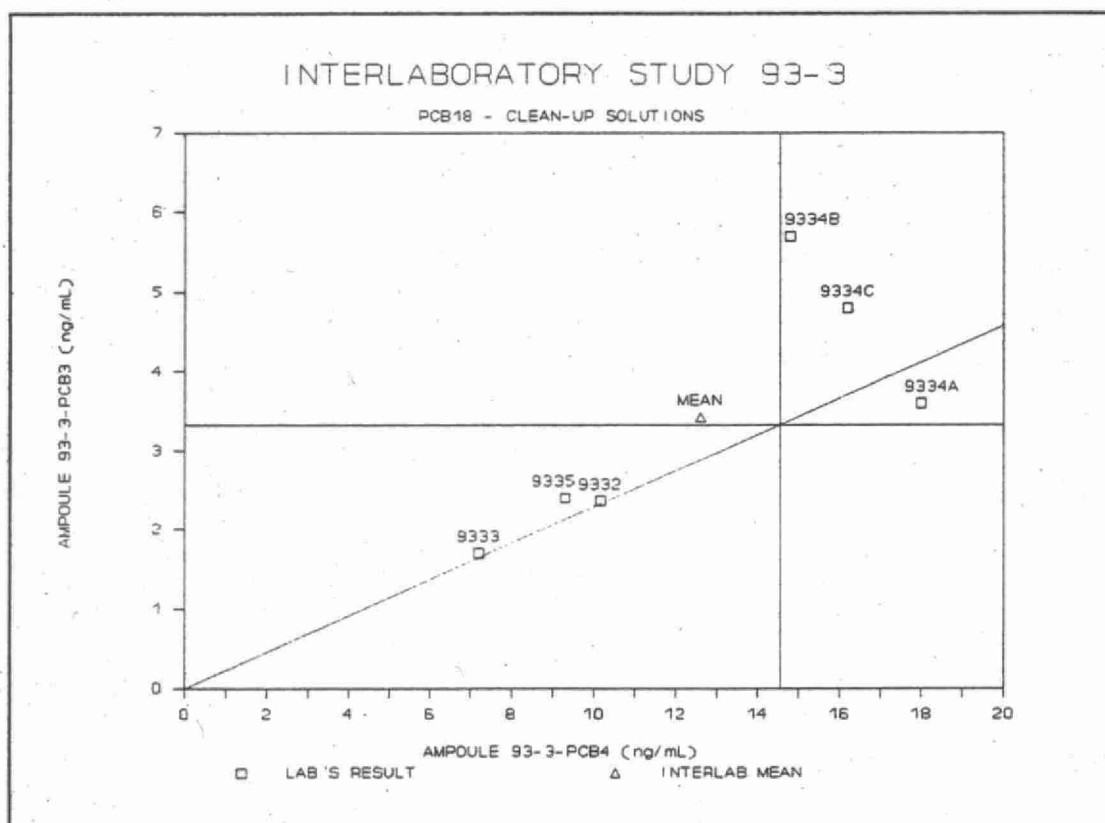


Figure 6 - PCB18 from Ampoules Processed Through Clean-up

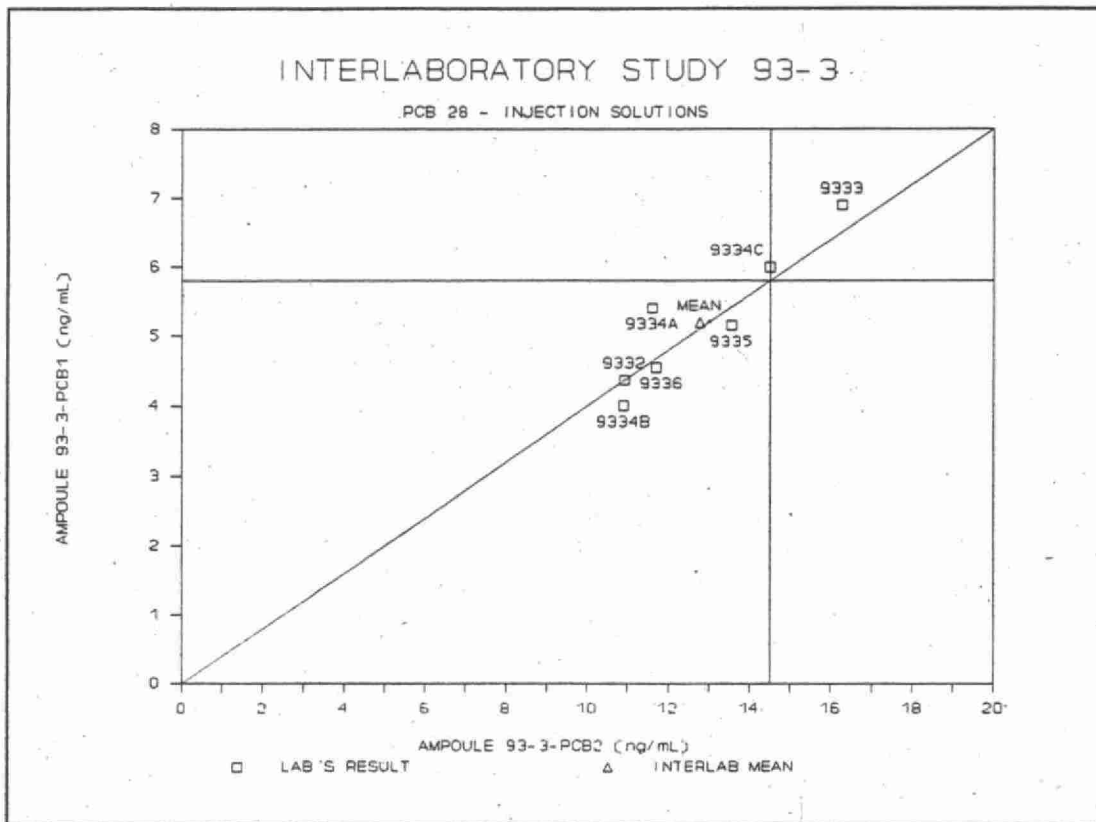


Figure 7 - PCB28 from Injection-Ready Ampoules

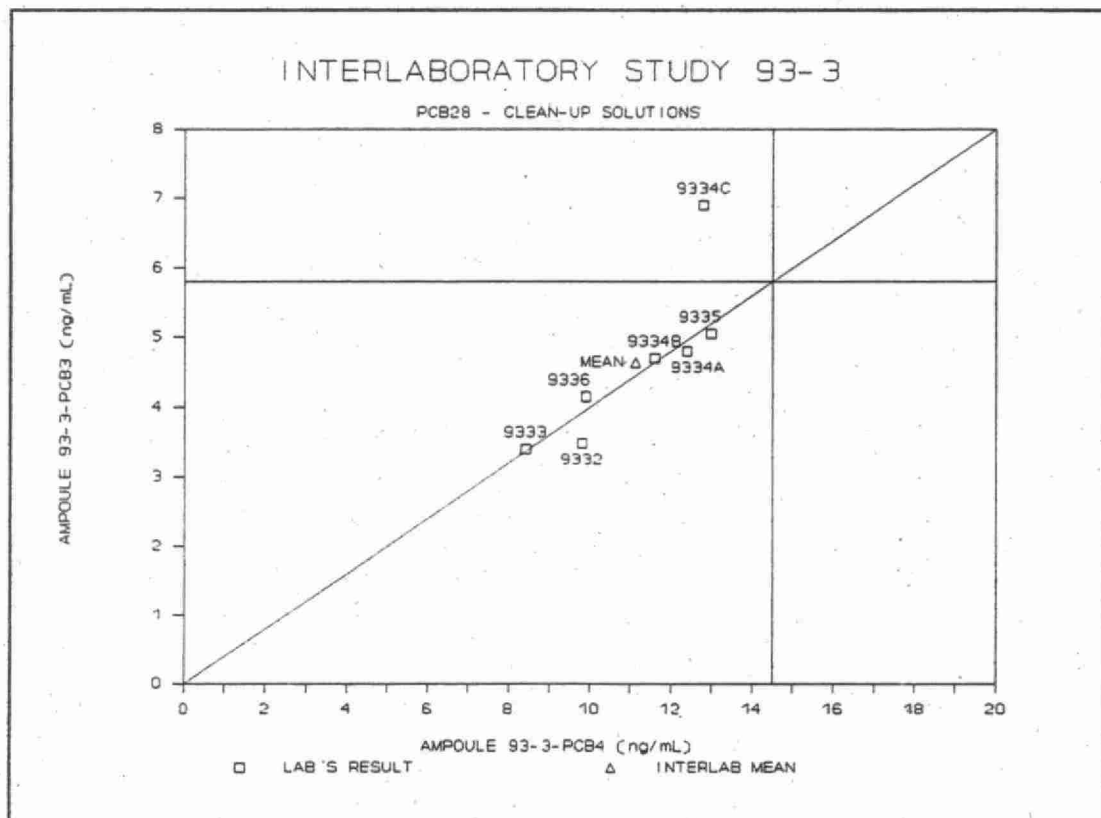


Figure 8 - PCB28 from Ampoules Processed through Clean-up

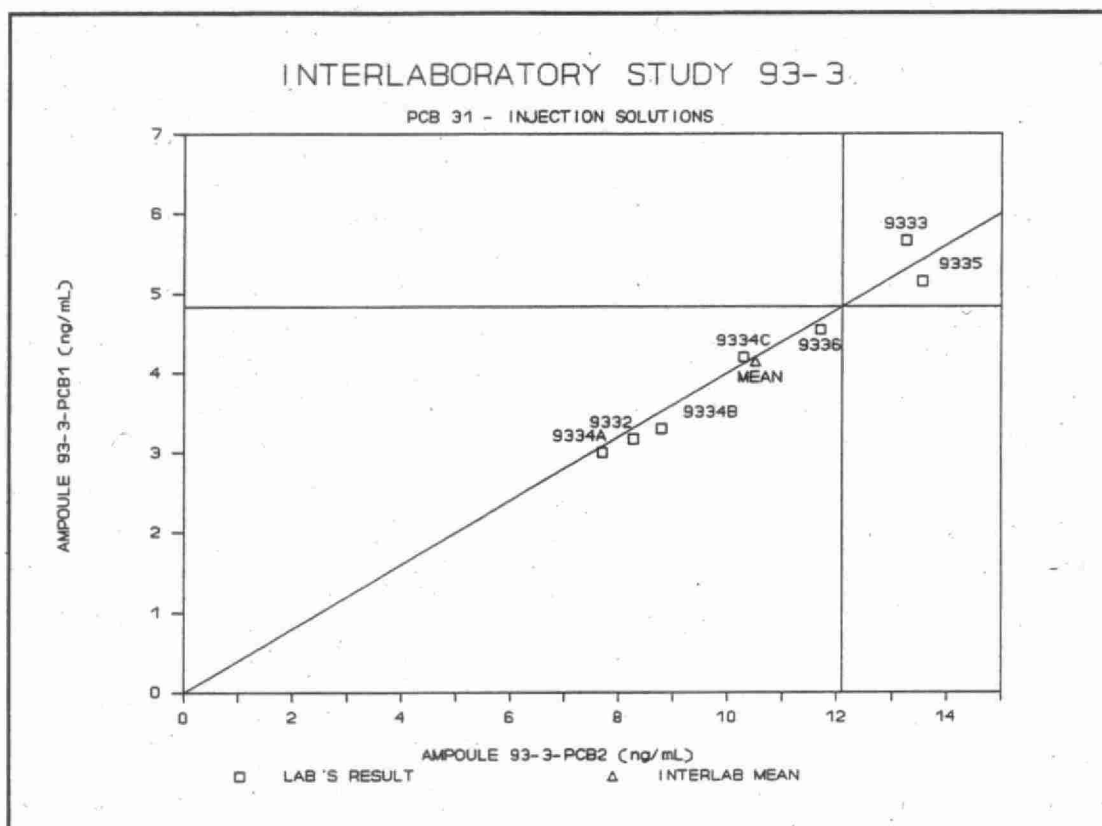


Figure 9 - PCB31 from Injection-Ready Ampoules

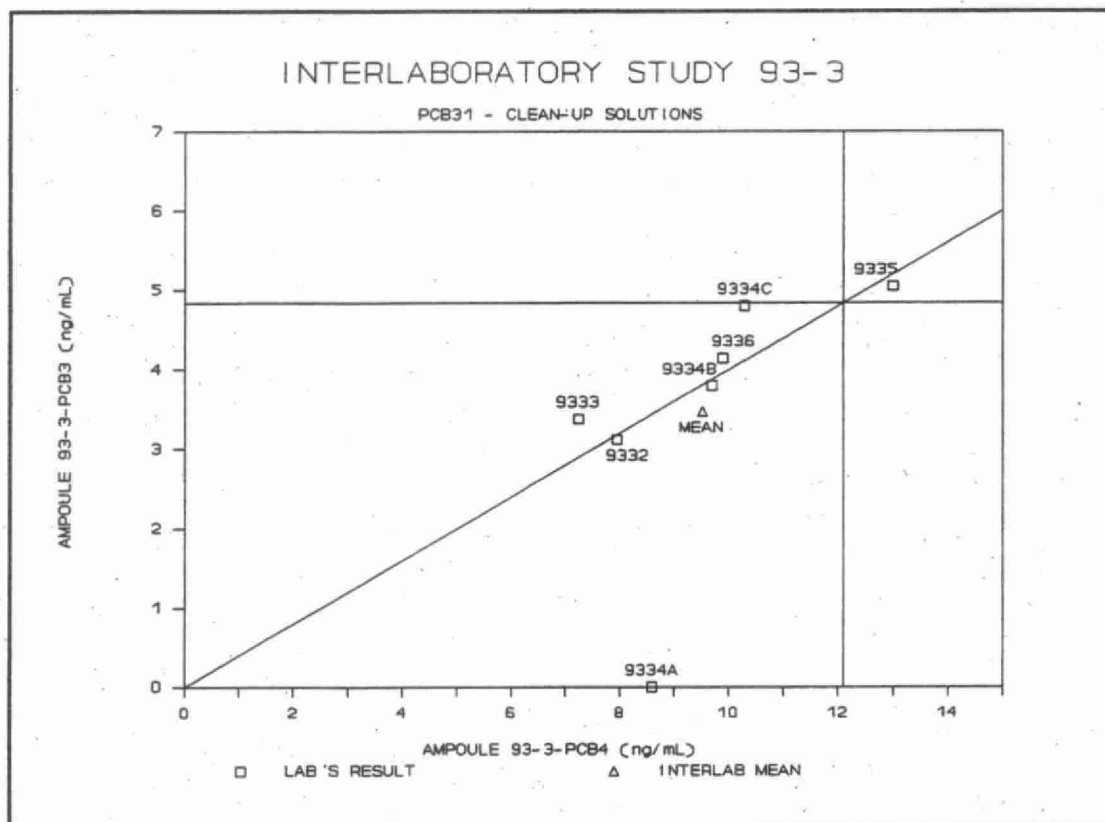


Figure 10 - PCB31 from Ampoules Processed through Clean-up

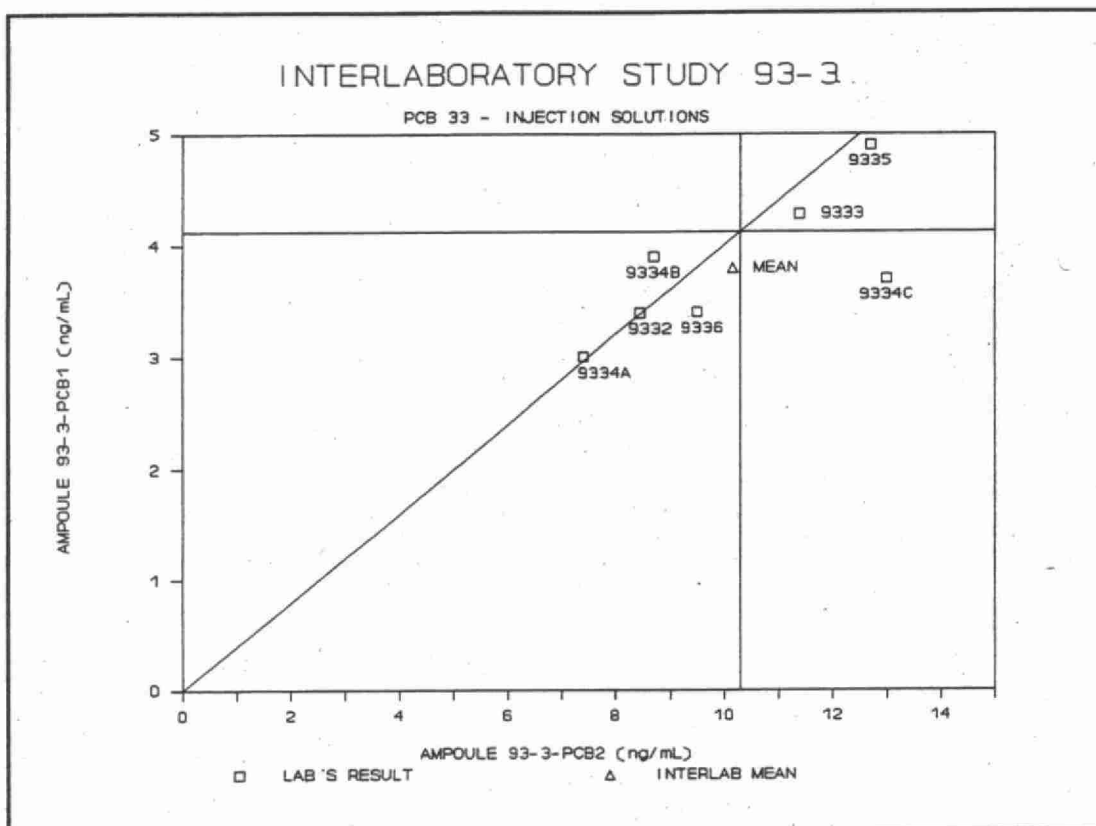


Figure 11 - PCB33 from Injection-Ready Ampoules

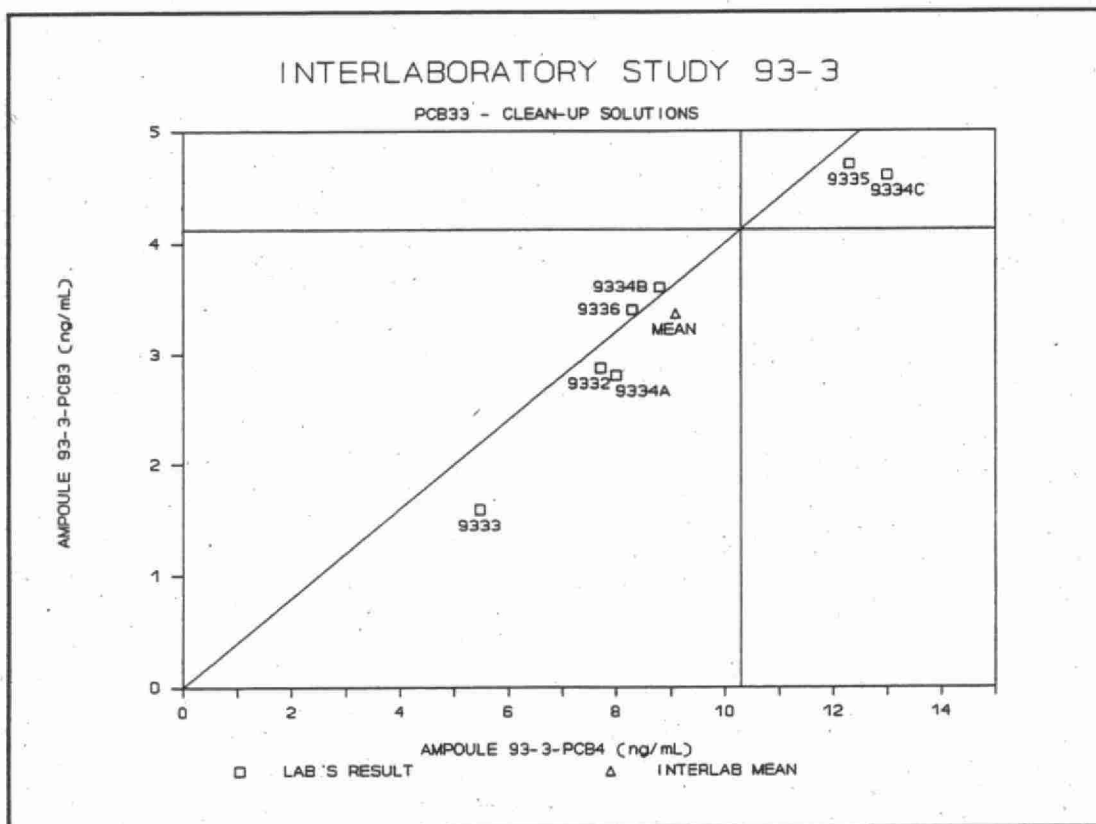


Figure 12 - PCB33 from Ampoules Processed through Clean-up

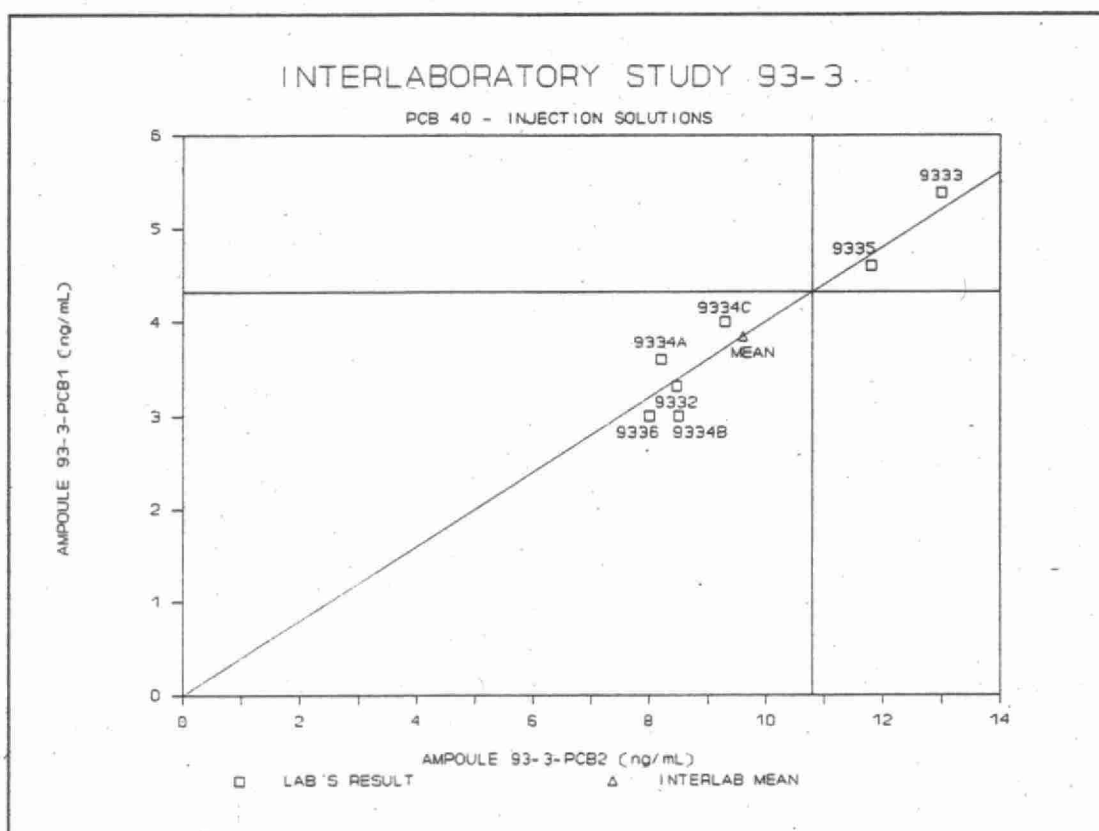


Figure 13 - PCB40 from Injection-Ready Ampoules

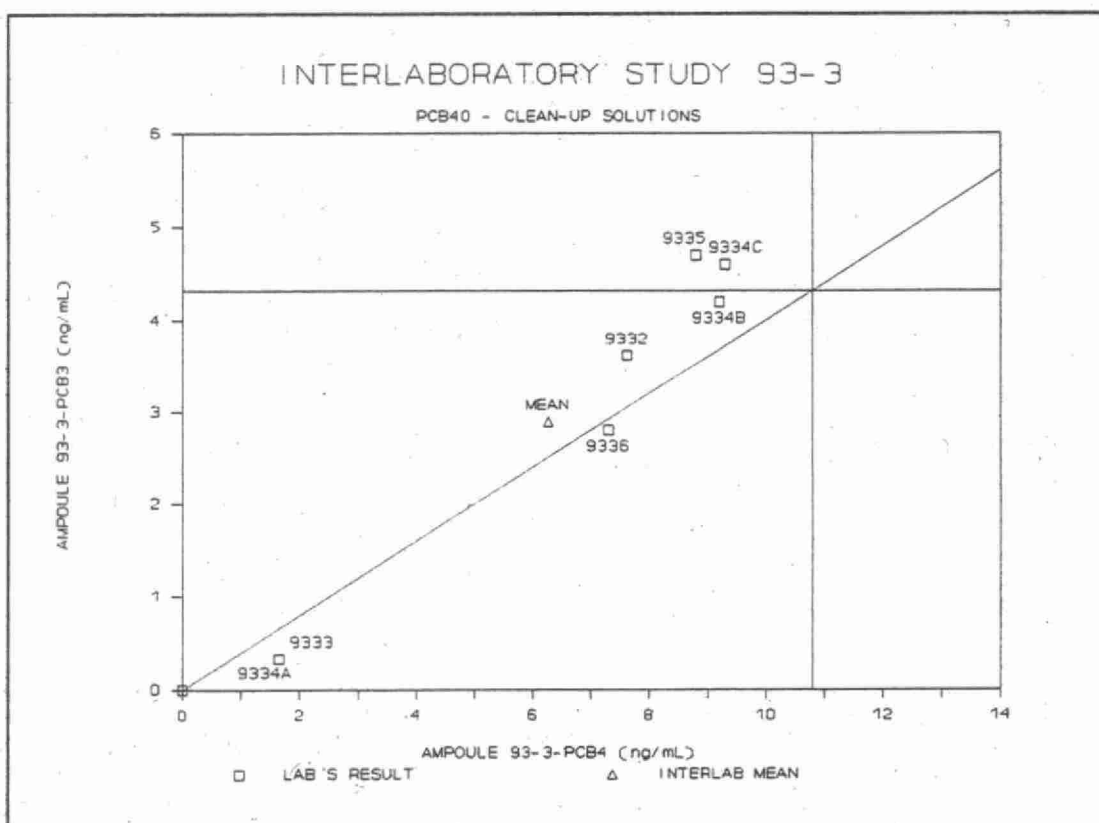


Figure 14 - PCB40 from Ampoules Processed through Clean-up

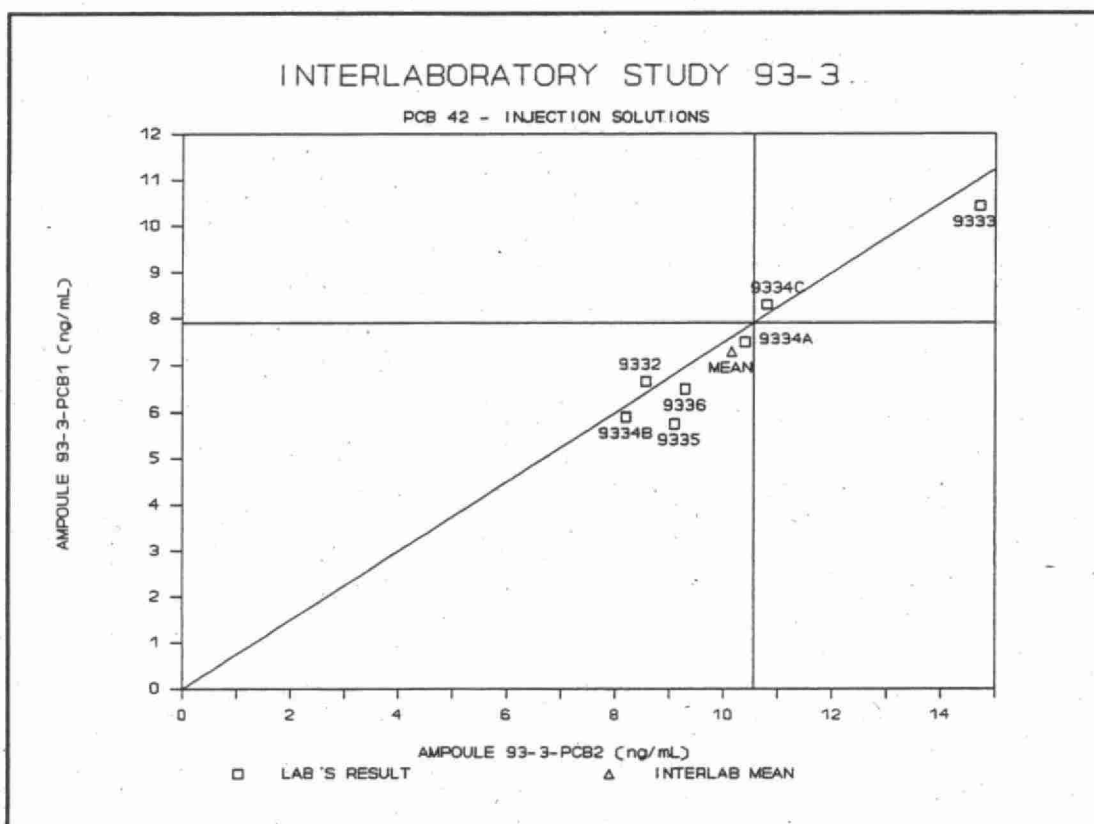


Figure 15 - PCB42 from Injection-Ready Solutions

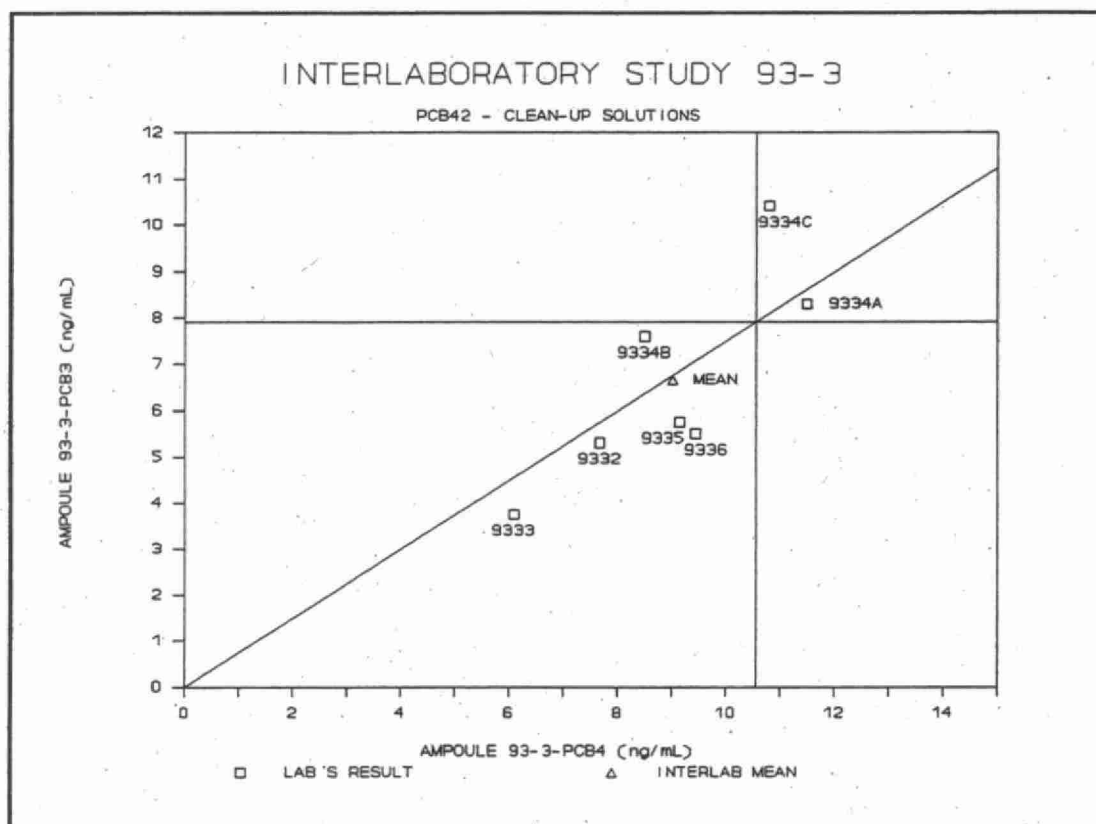


Figure 16 - PCB42 from Ampoules Processed through Clean-up

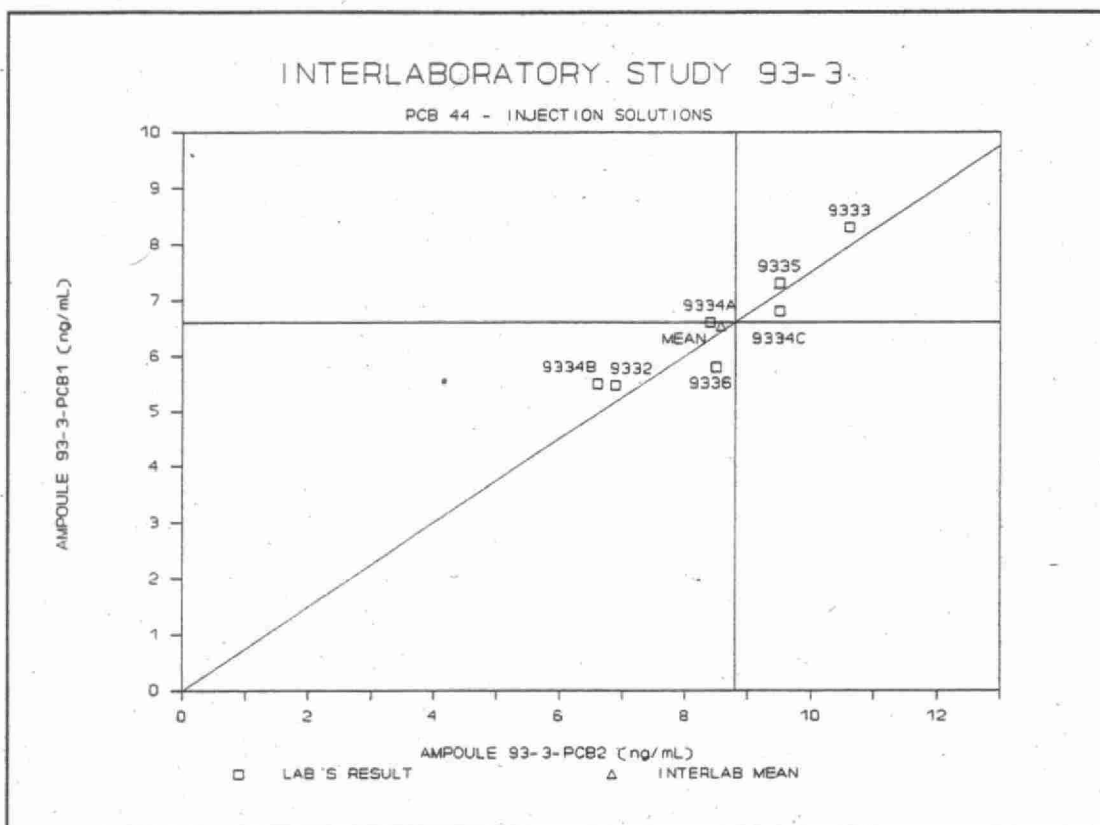


Figure 17 - PCB44 from Injection-Ready Ampoules

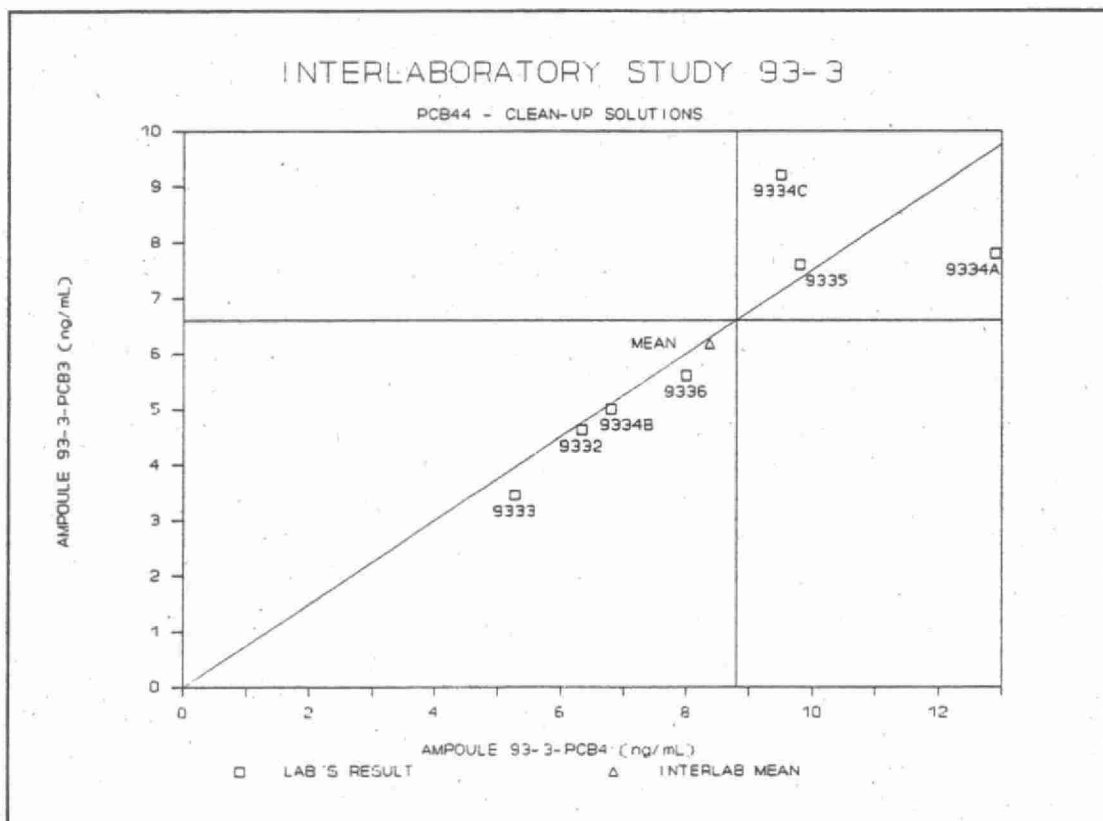


Figure 18 - PCB44 from Ampoules Processed through Clean-up

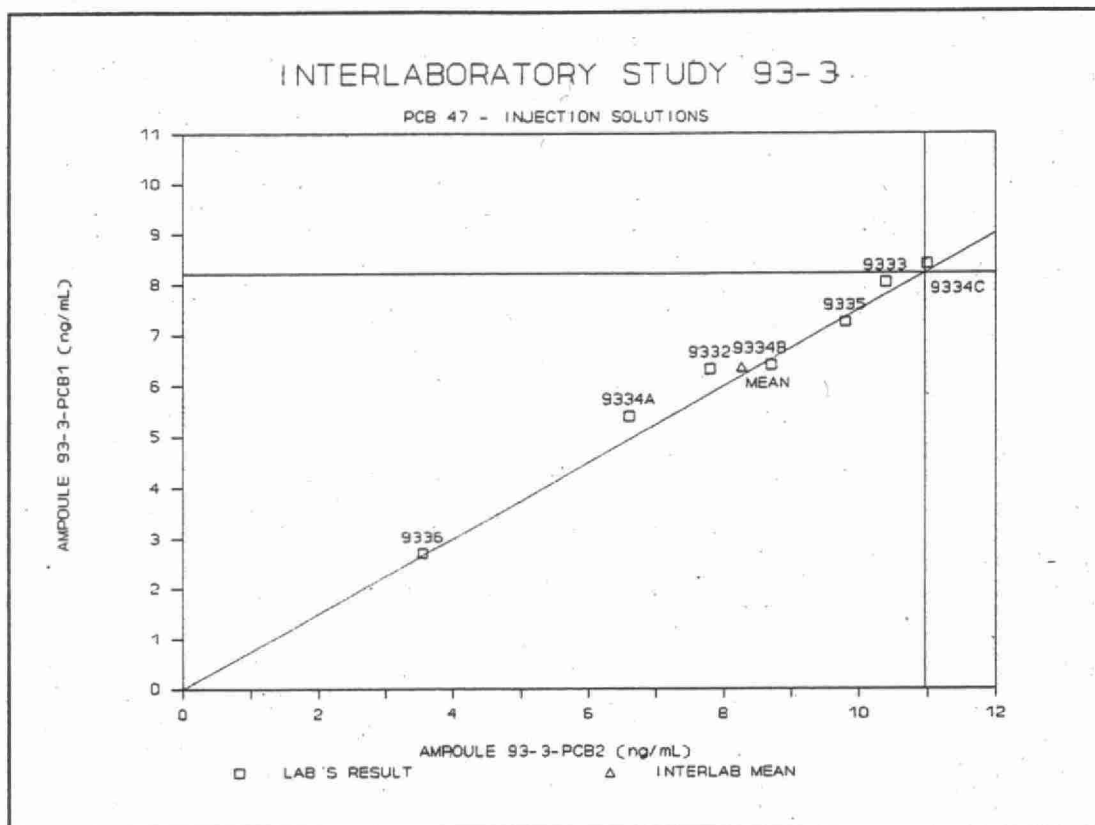


Figure 19 - PCB47 from Injection-Ready Ampoules

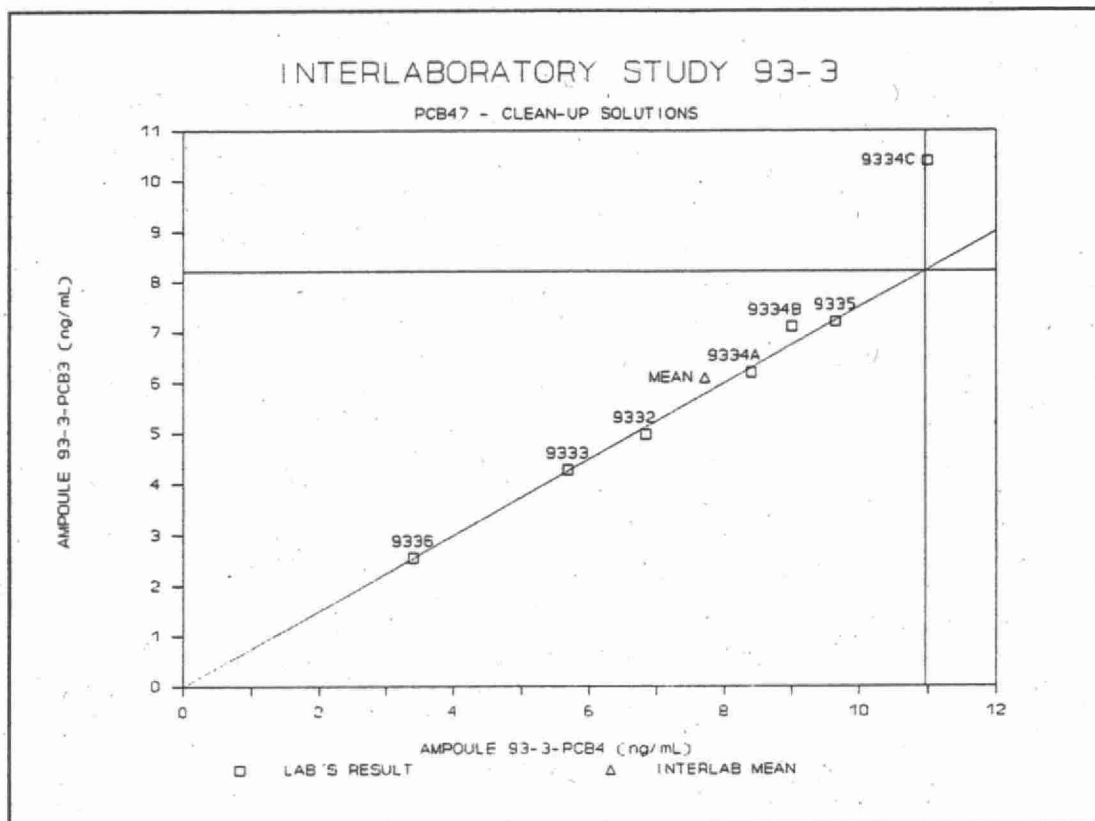


Figure 20 - PCB47 from ampoules Processed through Clean-up

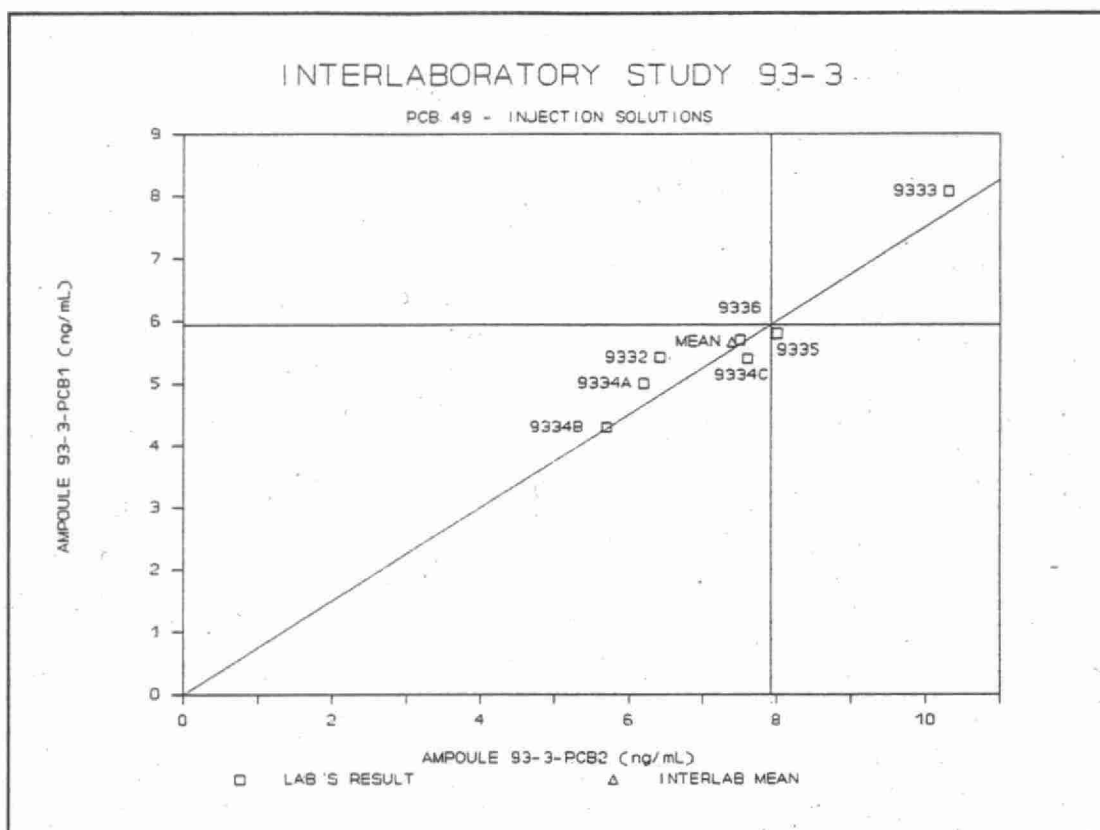


Figure 21 - PCB49 from Injection-Ready Ampoules

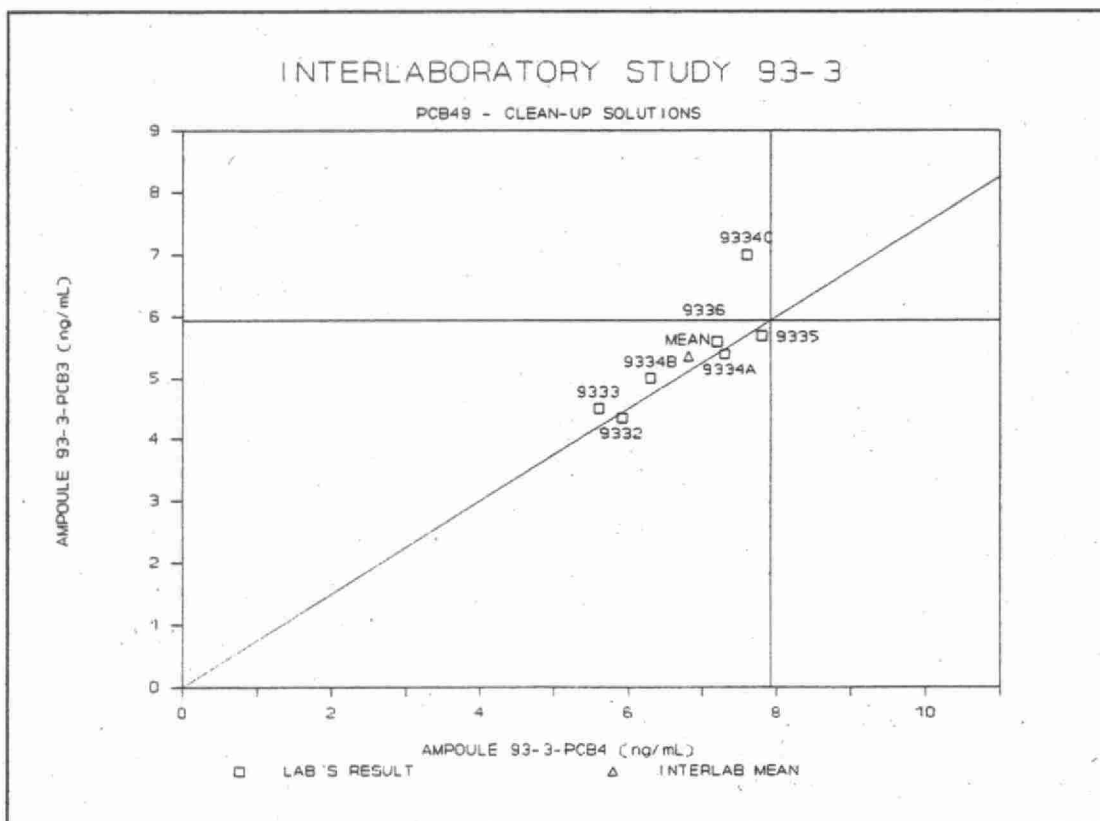


Figure 22 - PCB49 from Ampoules Processed through Clean-up

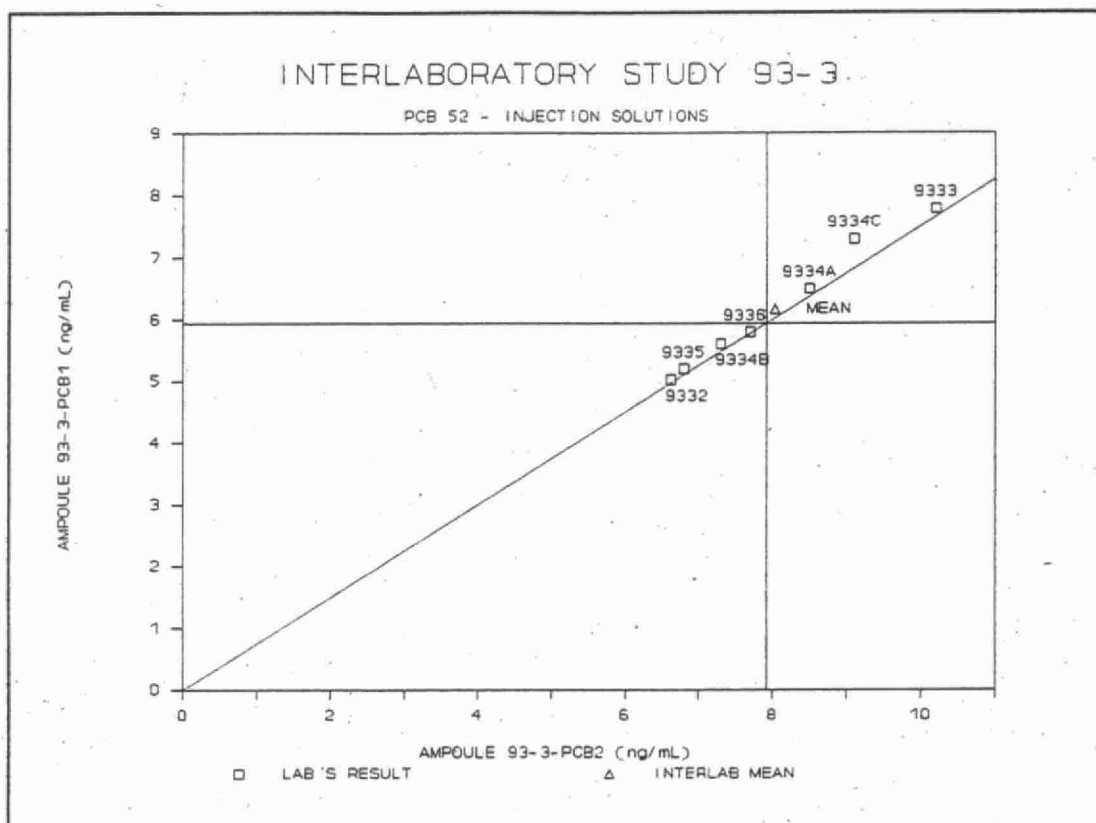


Figure 23 - PCB52 from Injection-Ready Ampoules

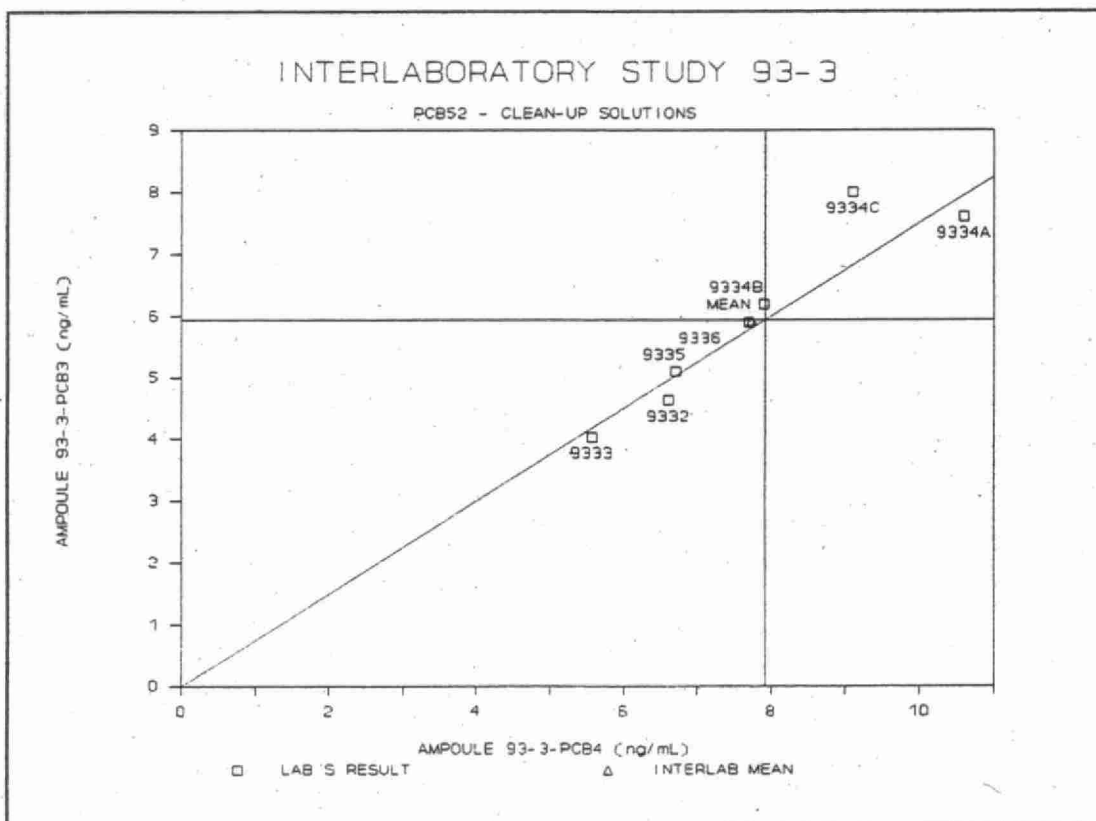


Figure 24 - PCB52 from Ampoules Processed through Clean-up

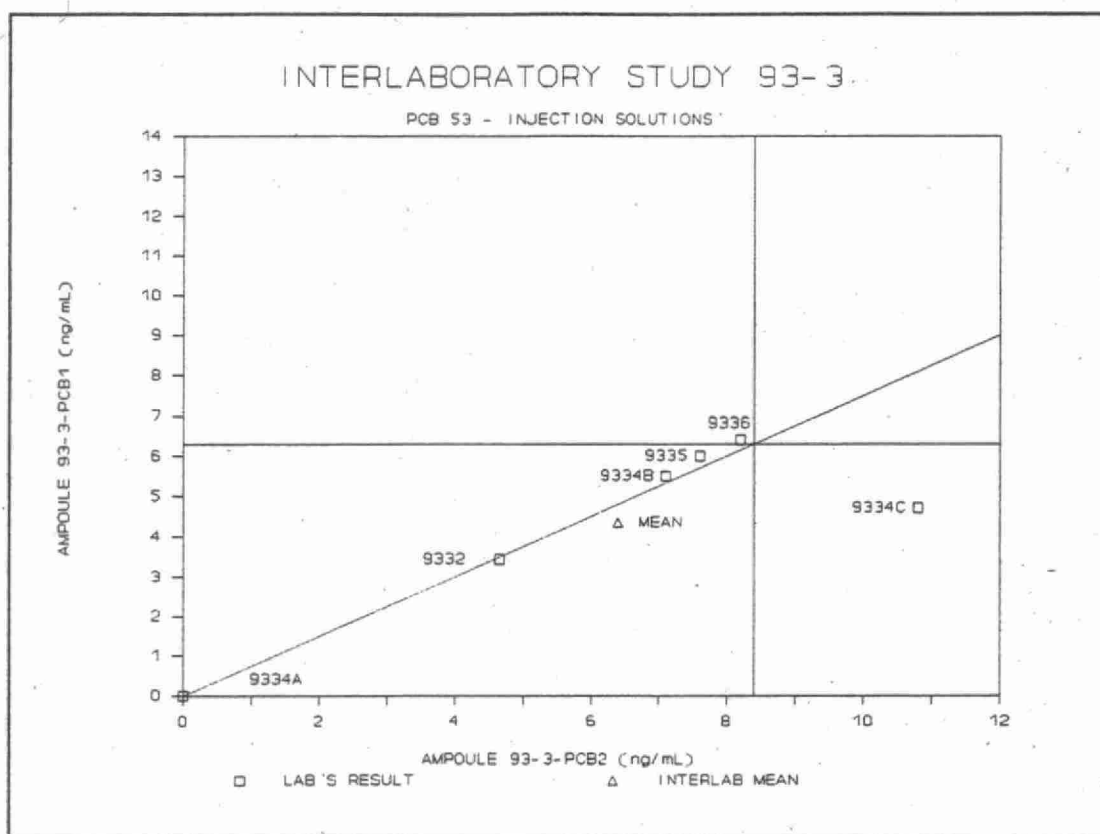


Figure 25 - PCB53 from Injection-Ready Ampoules

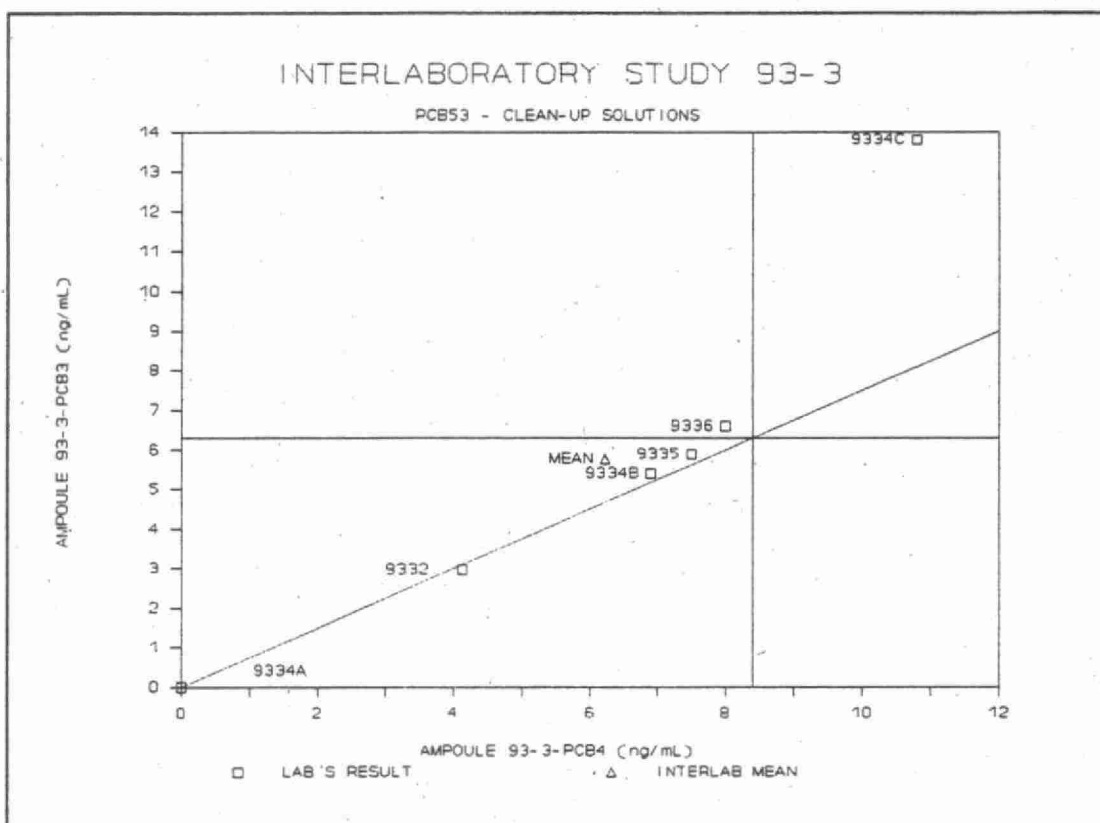


Figure 26 - PCB53 from Ampoules Processed through Clean-up

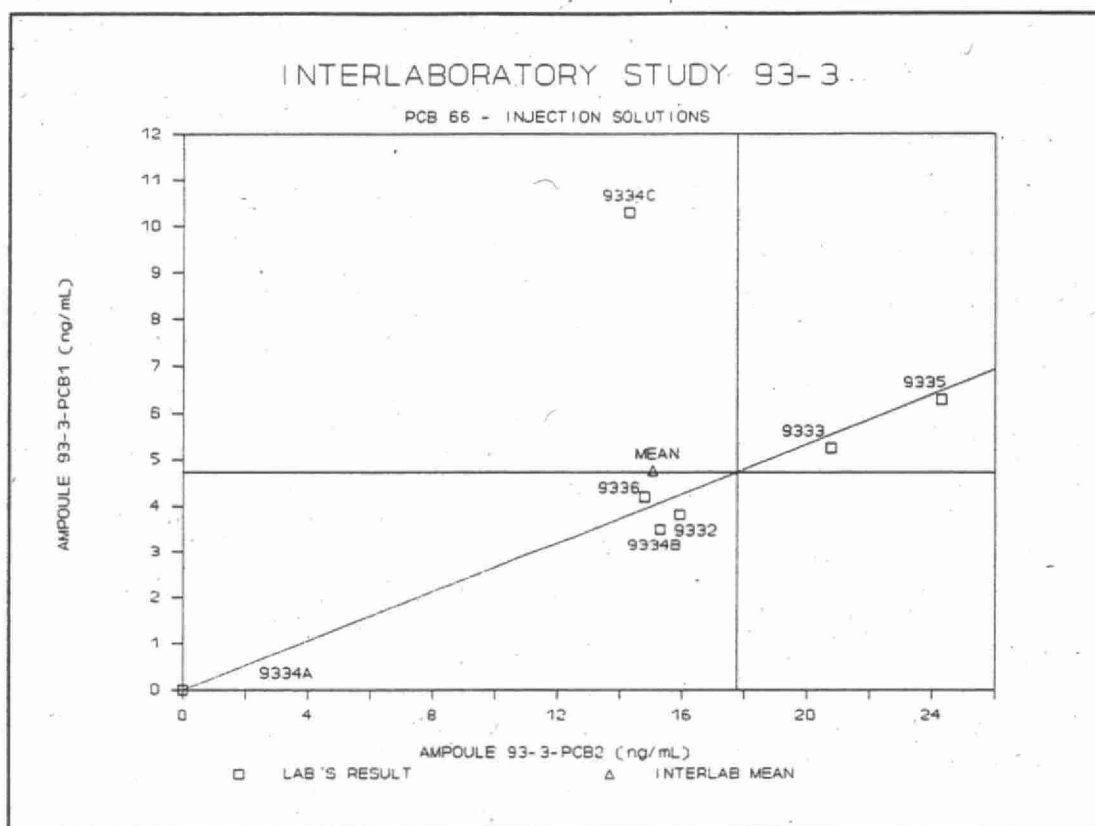


Figure 27 - PCB66 from Injection-Ready Ampoules

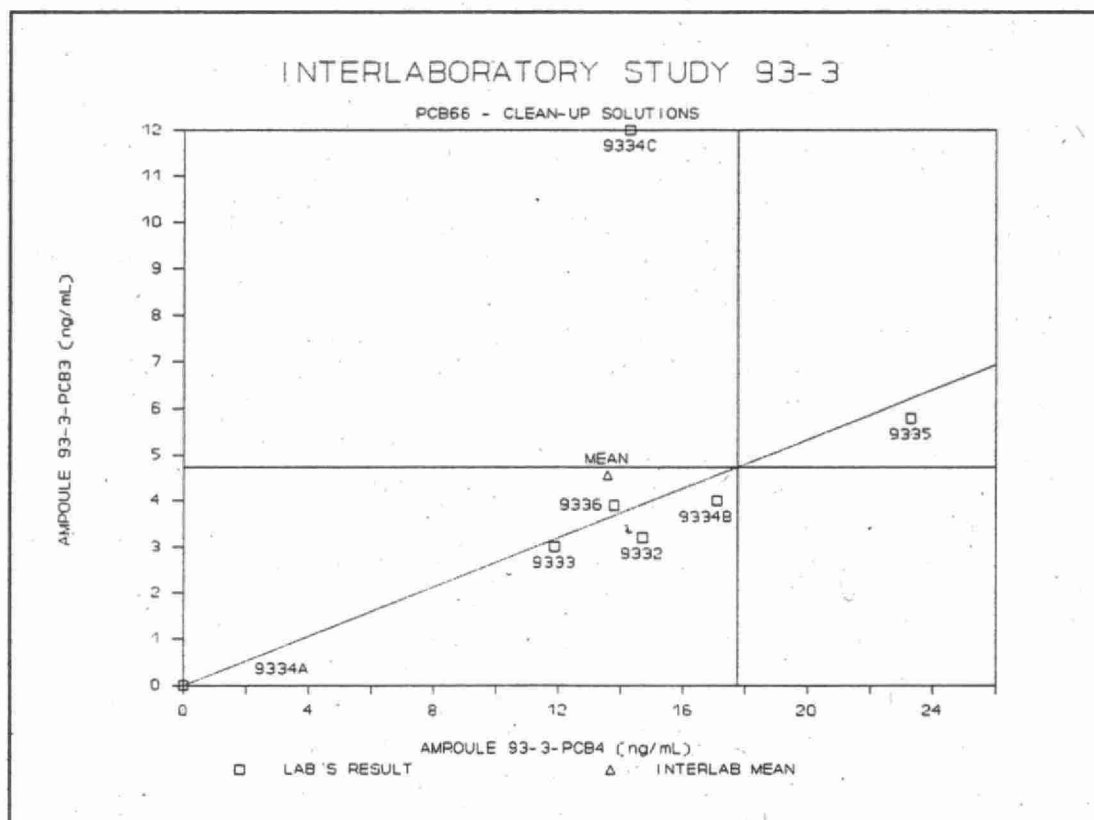


Figure 28 - PCB66 from Ampoules Processed through Clean-up

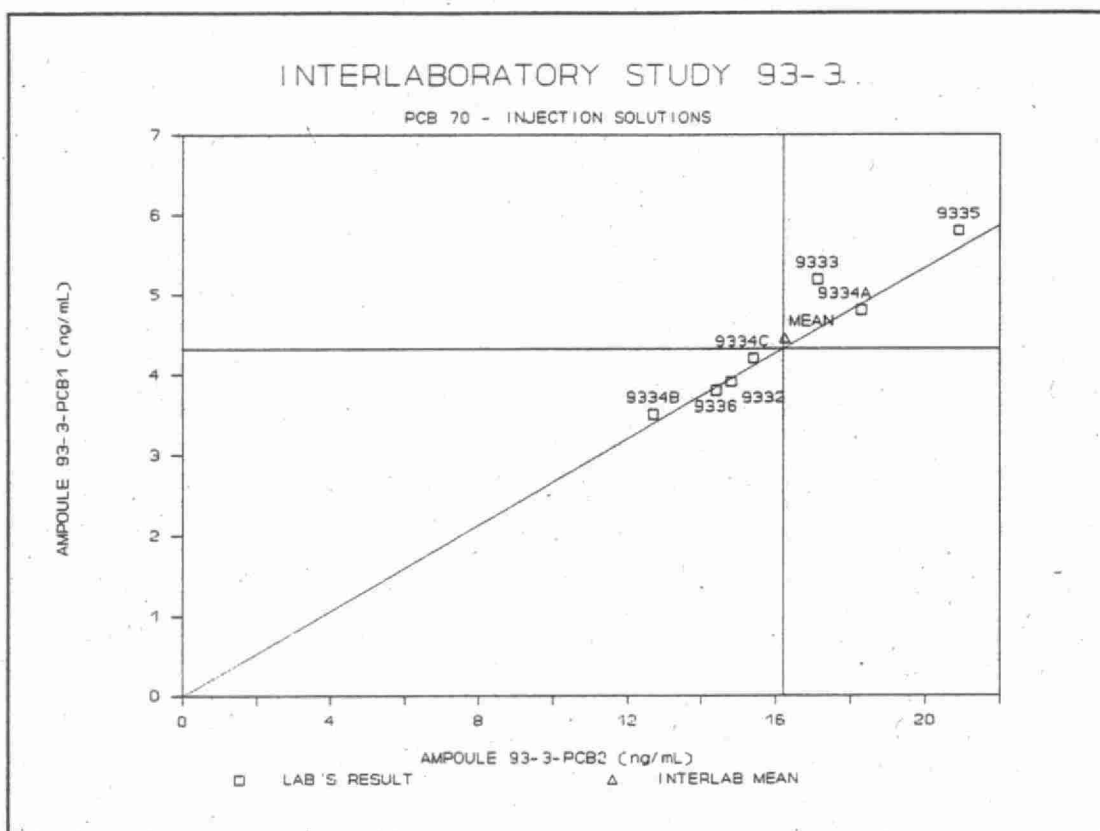


Figure 29 - PCB70 from Injection-Ready Ampoules

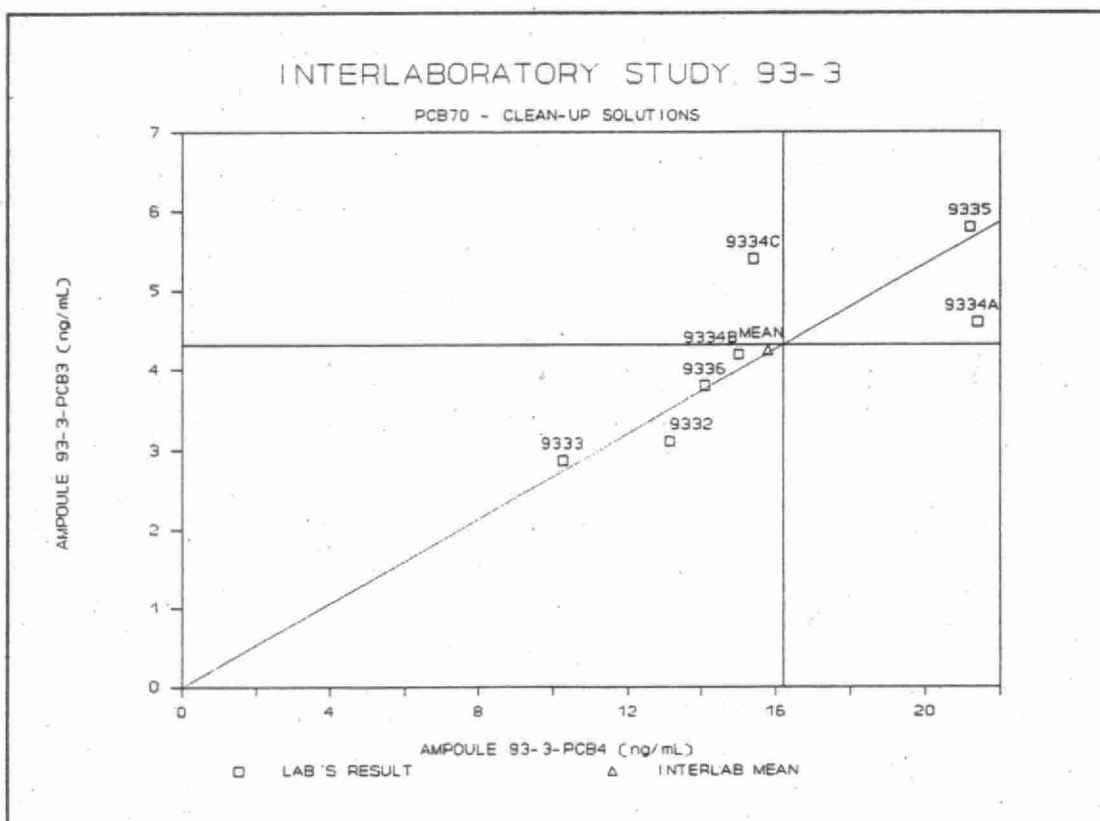


Figure 30 - PCB70 from Ampoules Processed through Clean-up

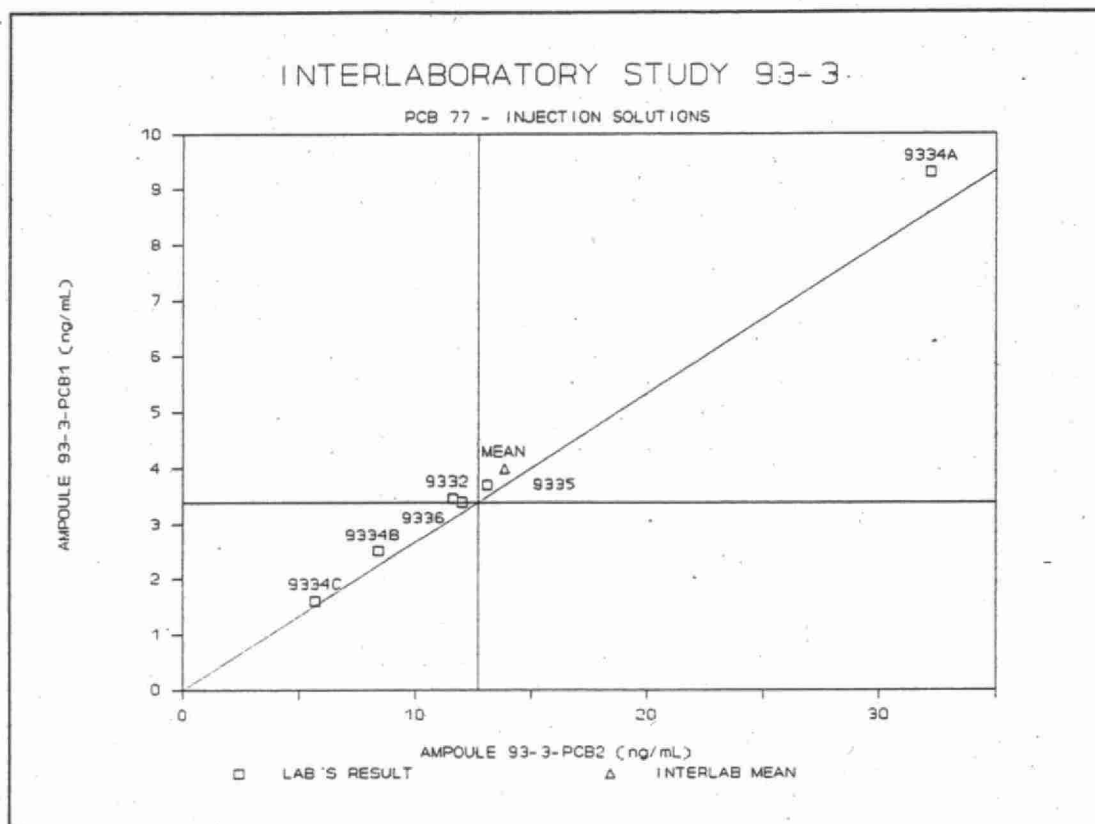


Figure 31 - PCB77 from Injection-Ready Ampoules

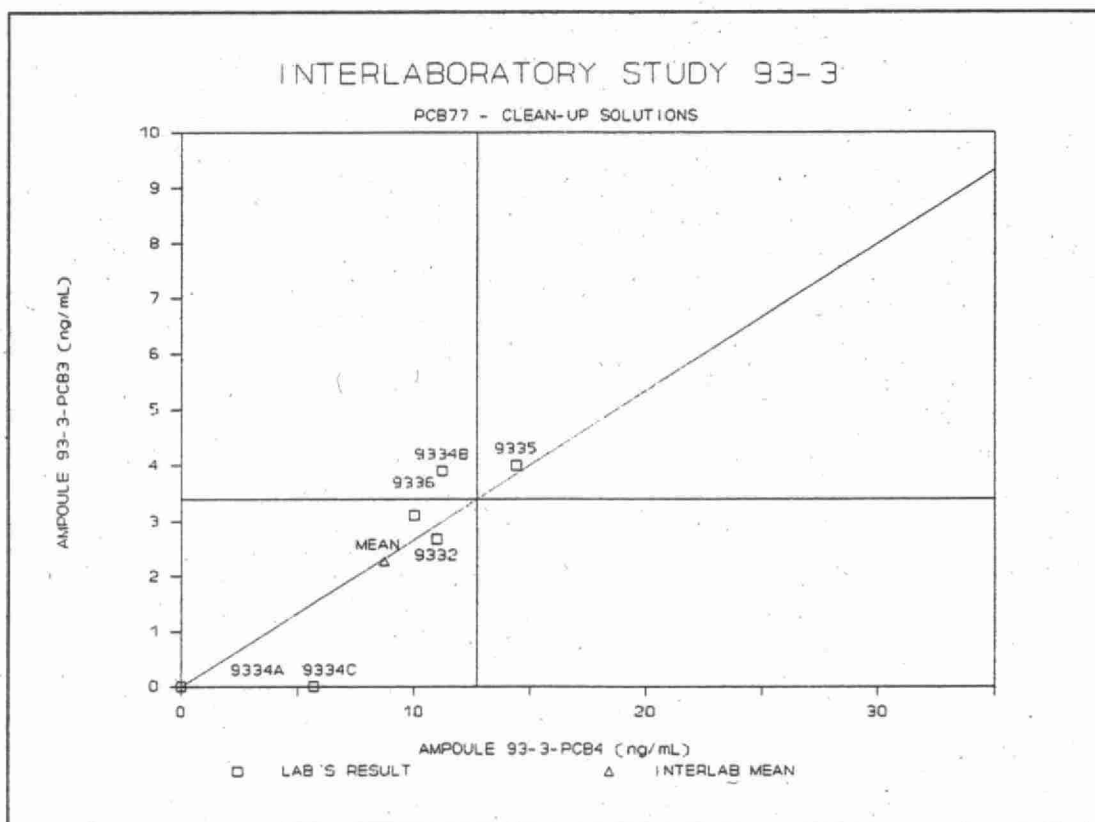


Figure 32 - PCB77 from Ampoules Processed through Clean-up

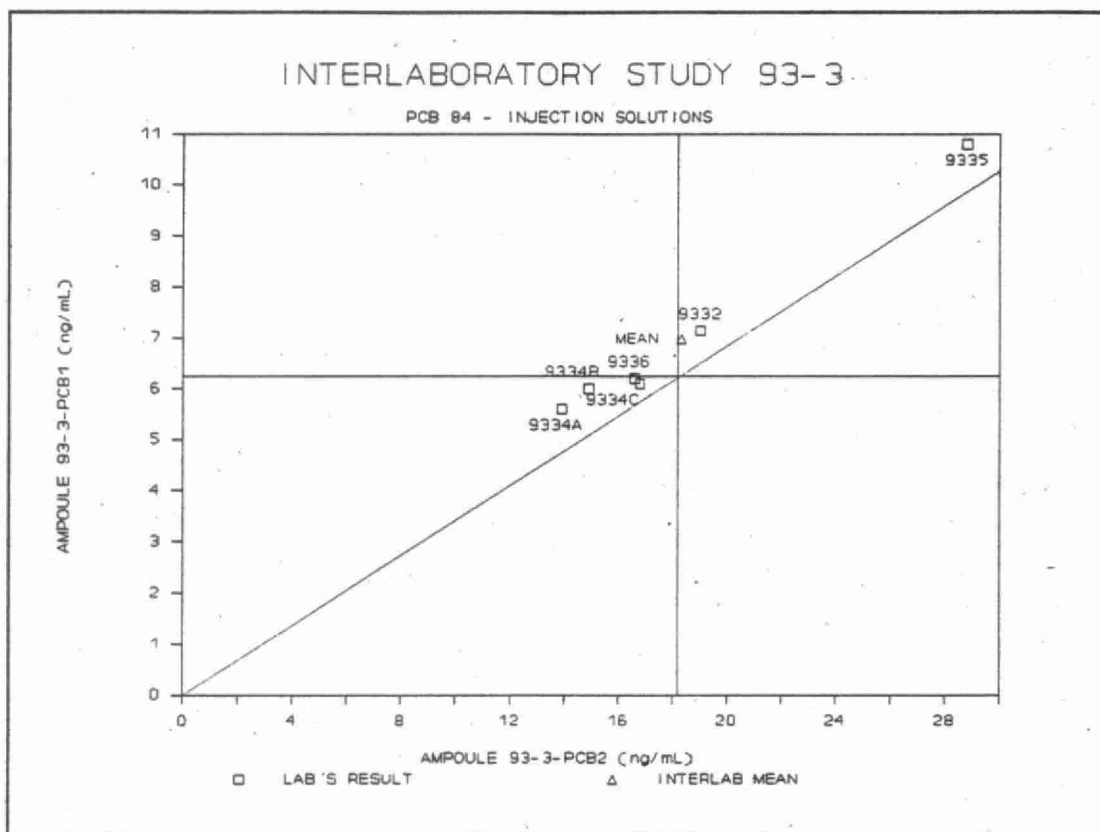


Figure 33 - PCB84 from Injection-Ready Ampoules

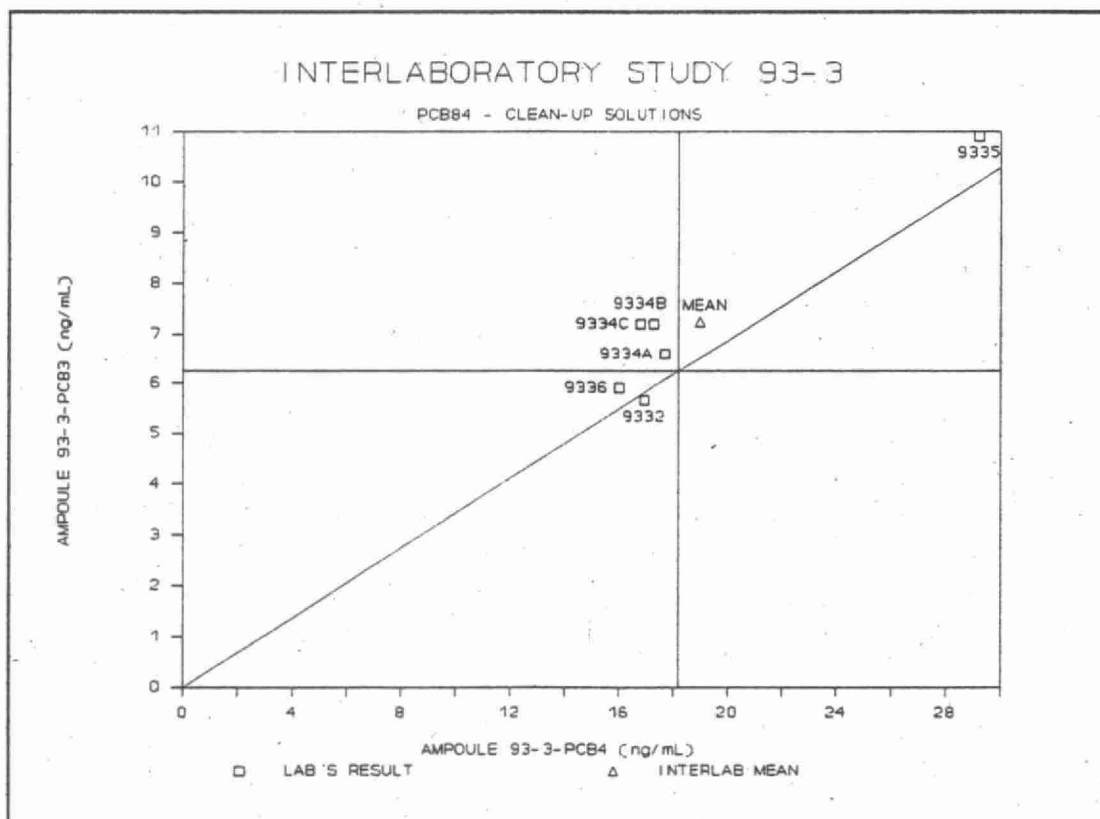


Figure 34 - PCB84 from Ampoules Processed through Clean-up

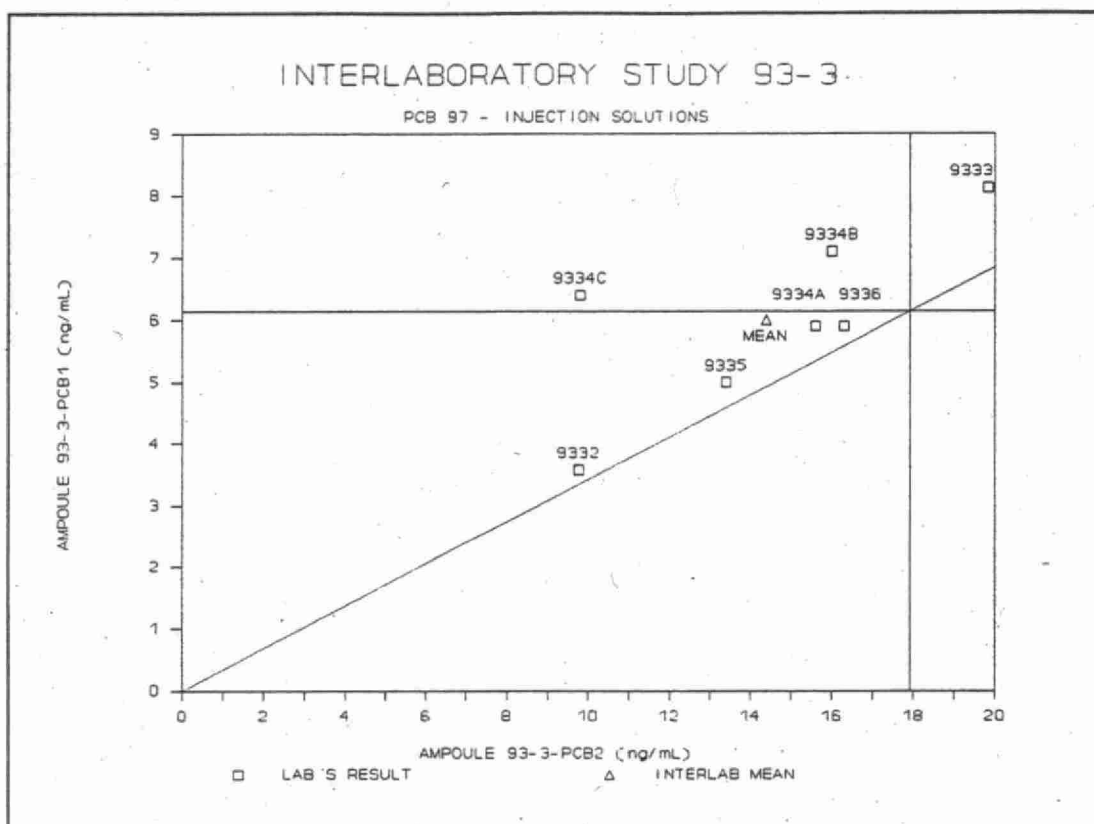


Figure 35 - PCB97 from Injection-Ready Ampoules

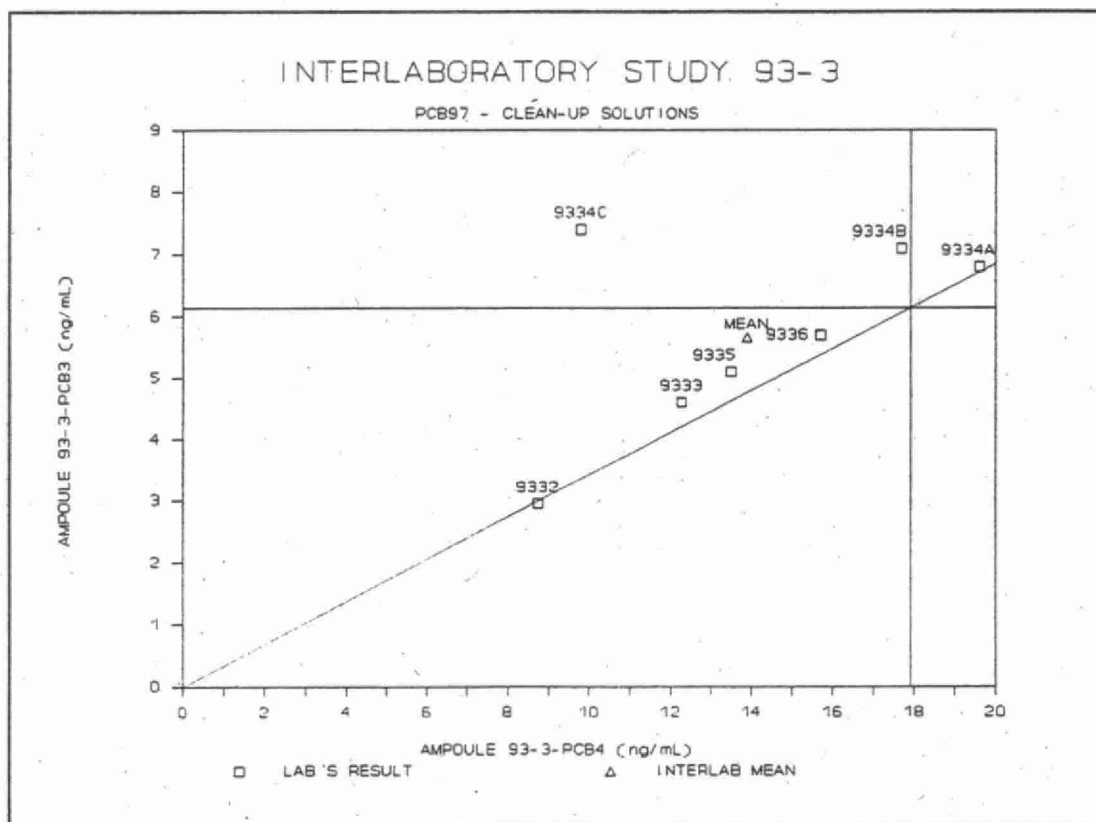


Figure 36 - PCB97 from Ampoules Processed through Clean-up

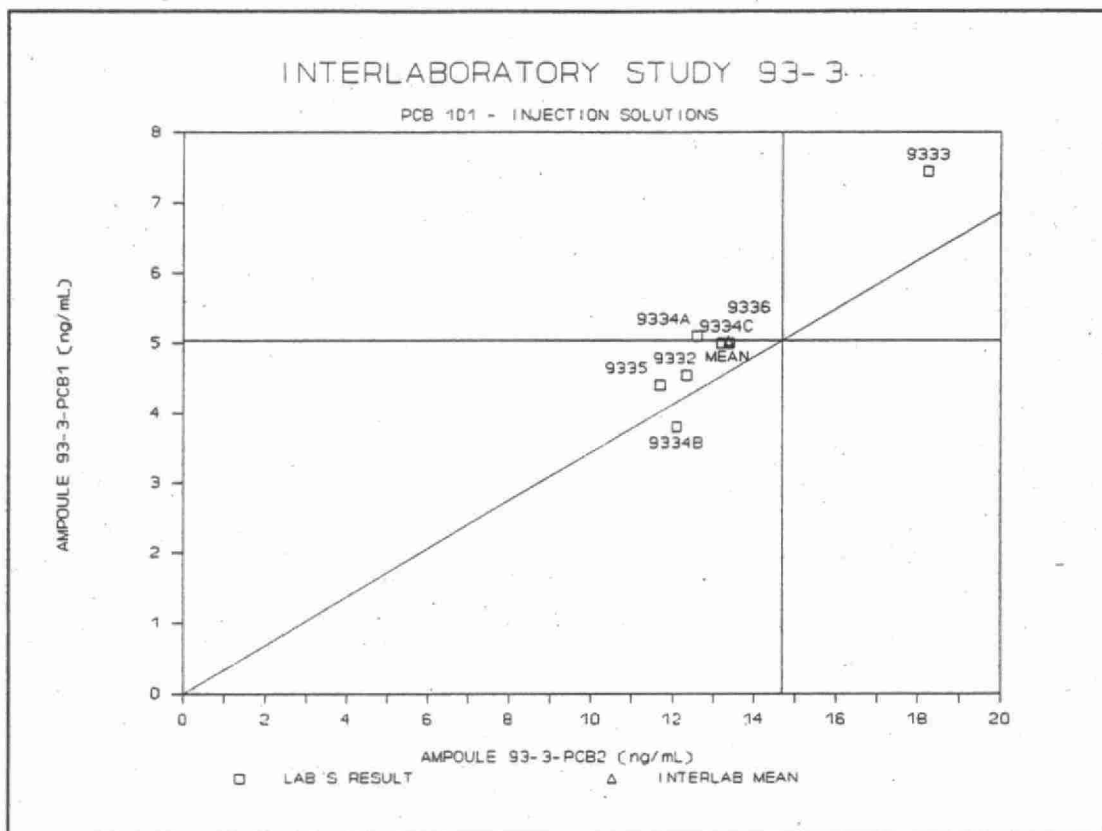


Figure 37 - PCB101 from Injection-Ready Ampoules

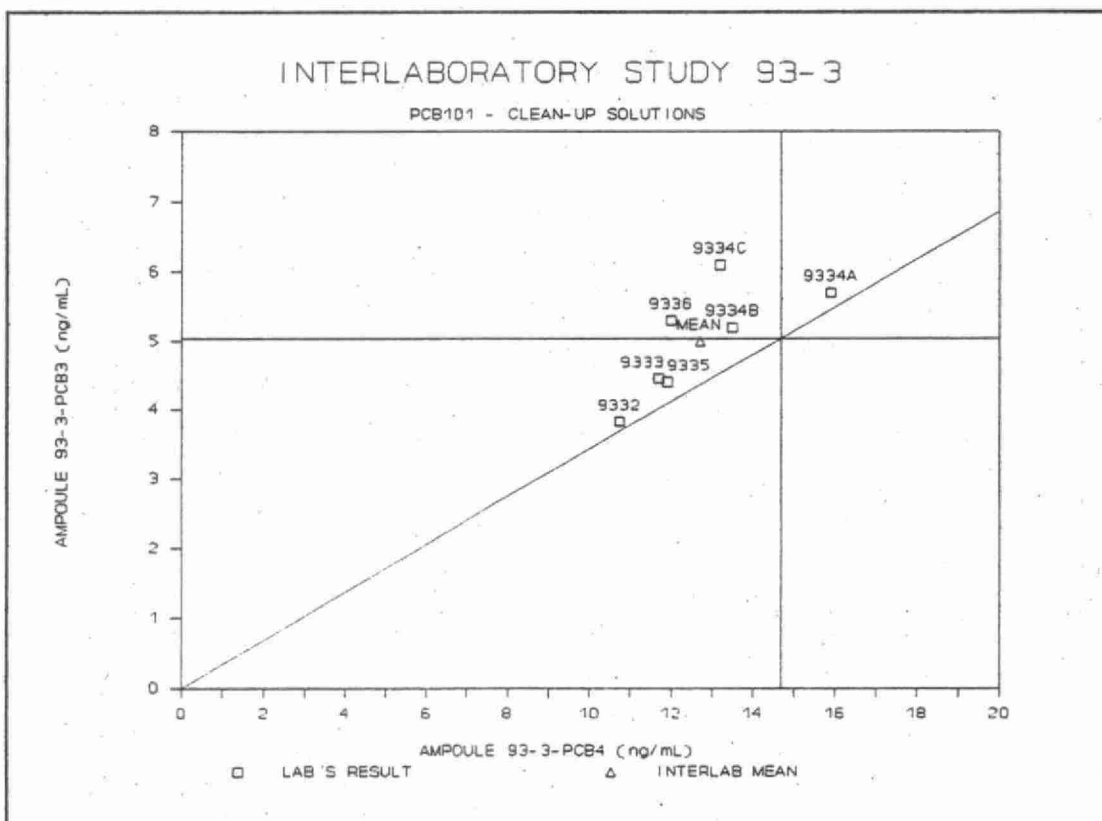


Figure 38 - PCB101 from Ampoules Processed through Clean-up

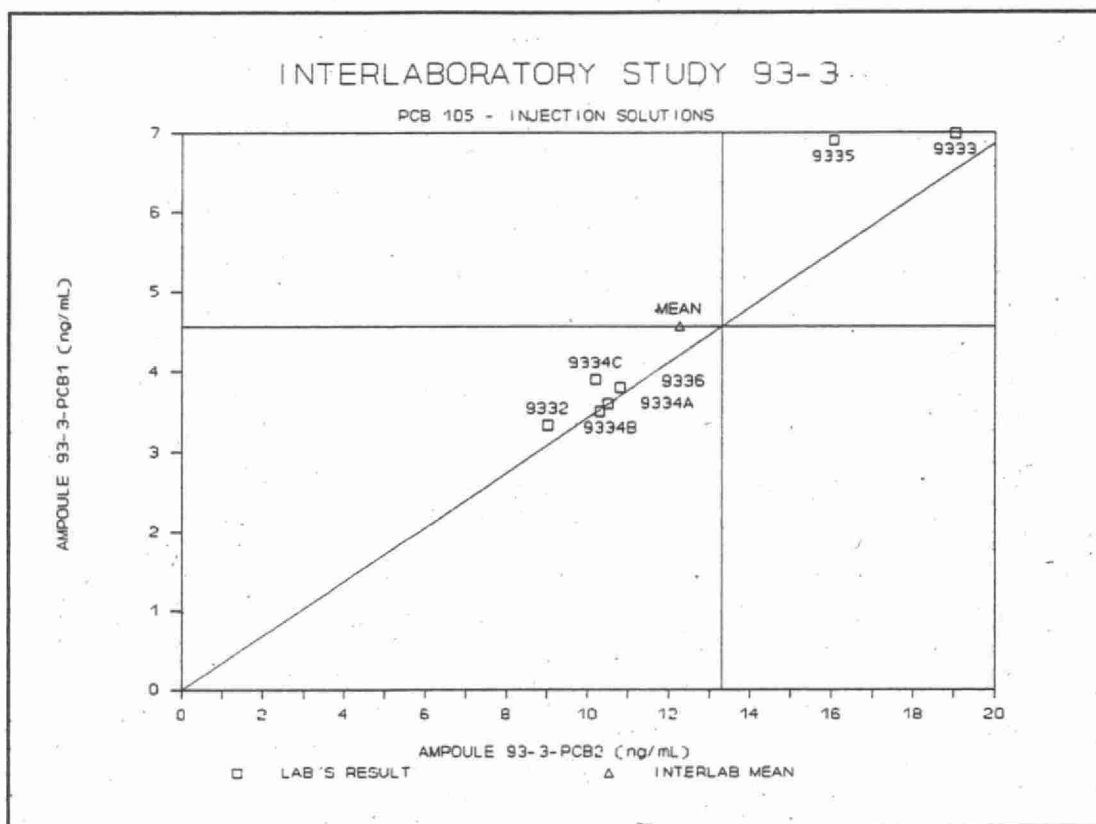


Figure 39 - PCB105 from Injection-Ready Ampoules

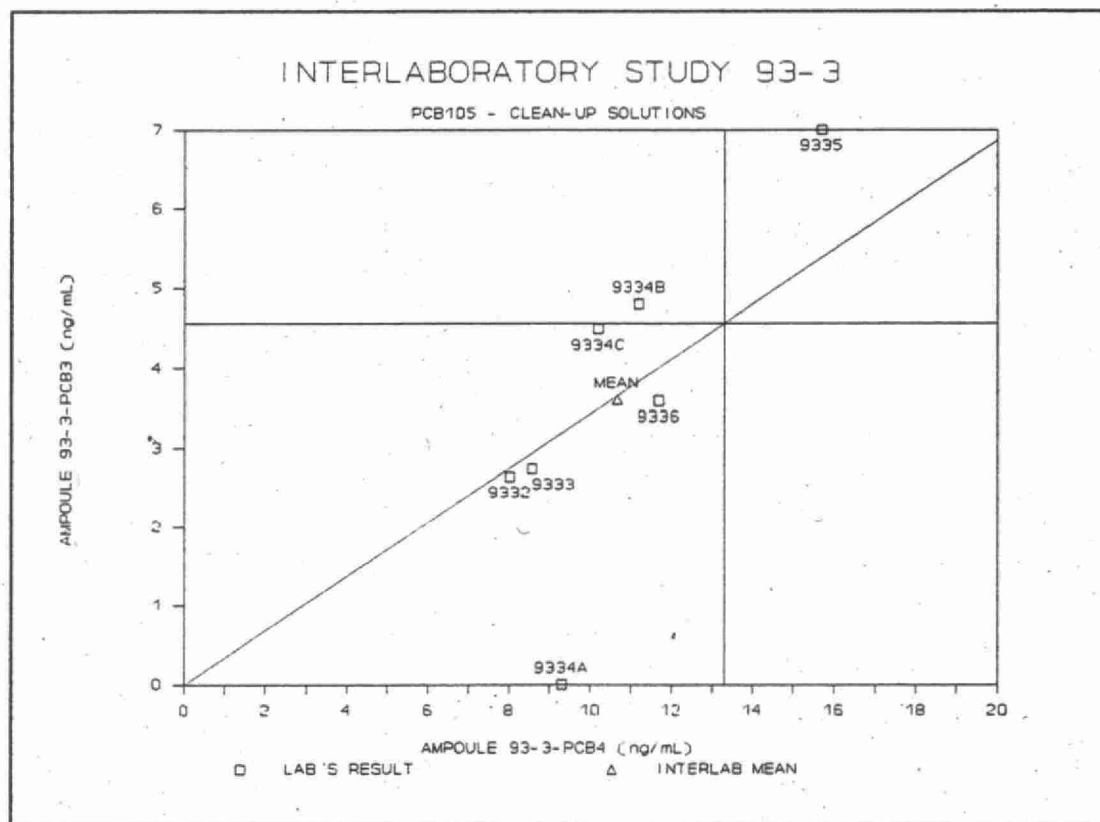


Figure 40 - PCB105 from Ampoules Processed through Clean-up

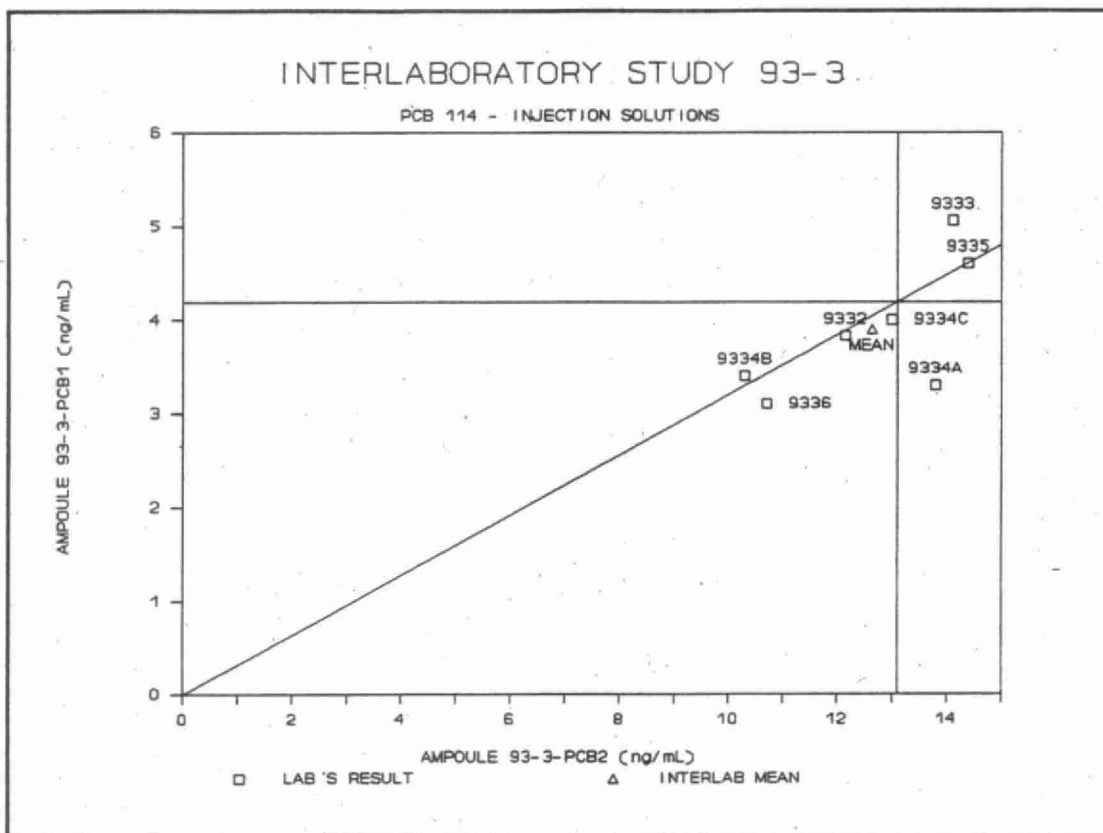


Figure 41 - PCB114 from Injection-Ready Ampoules

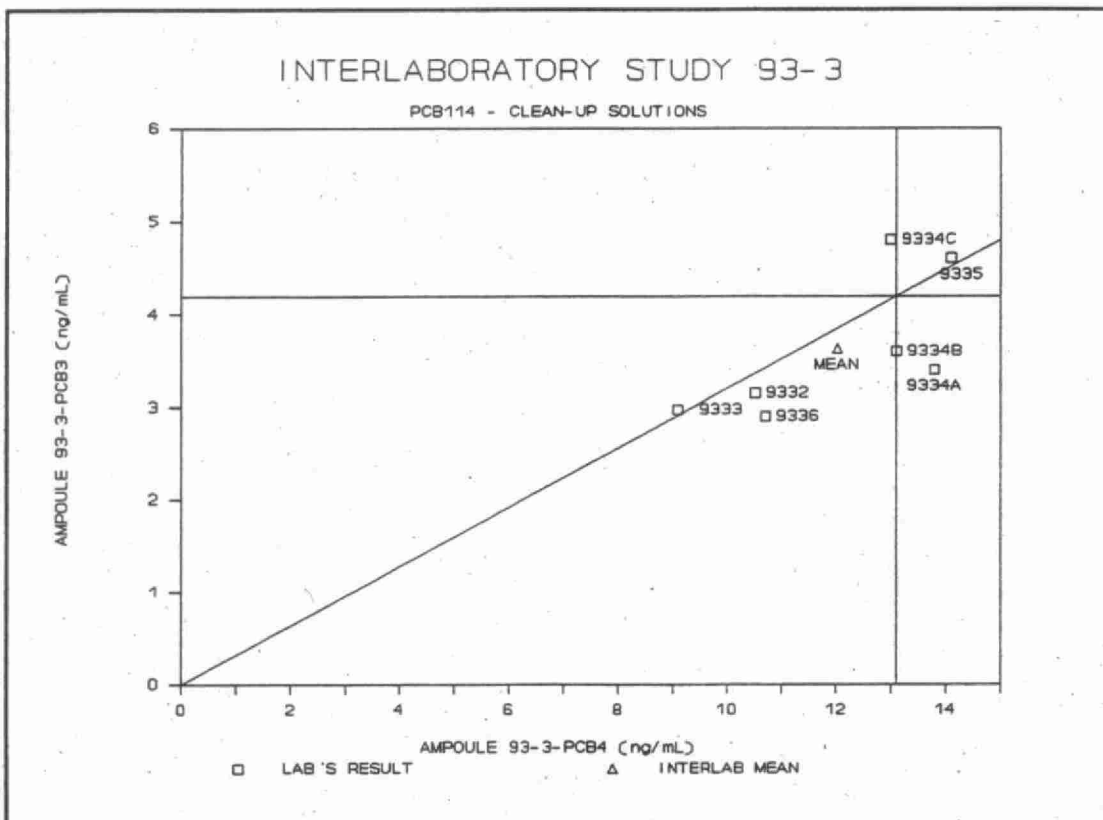


Figure 42 - PCB114 from Ampoules Processed through Clean-up

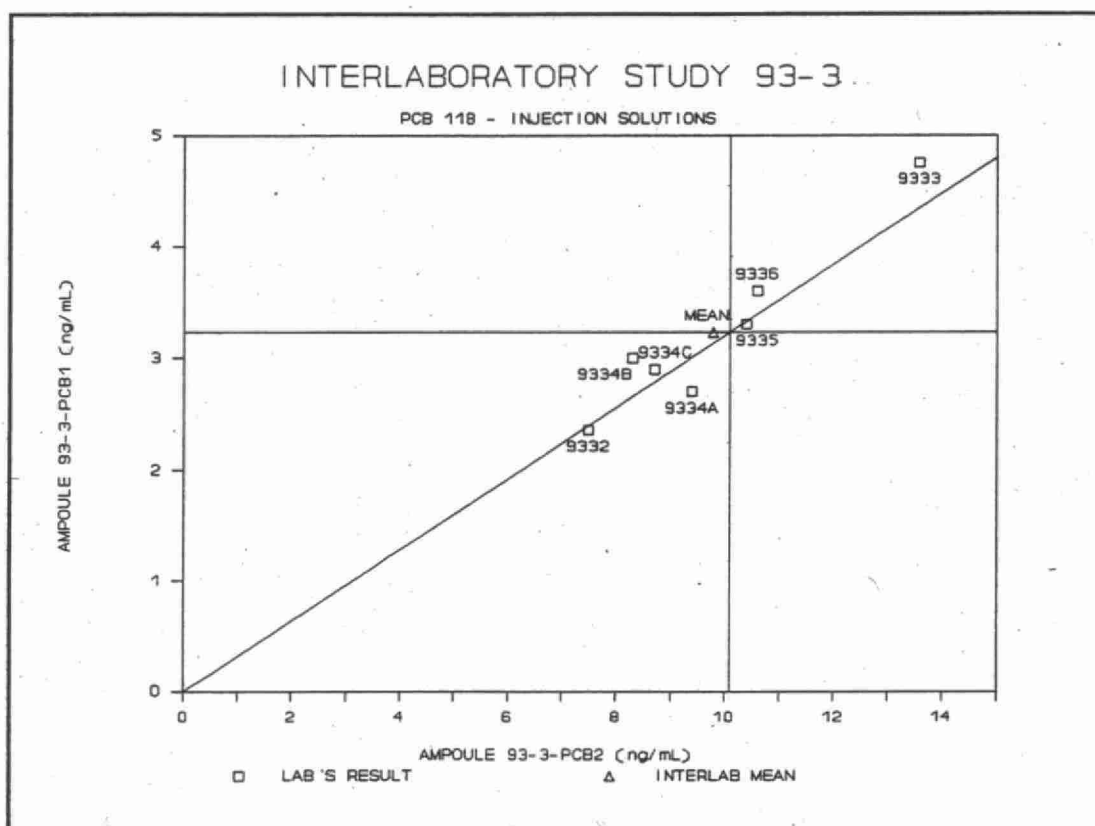


Figure 43 - PCB118 from Injection-Ready Ampoules

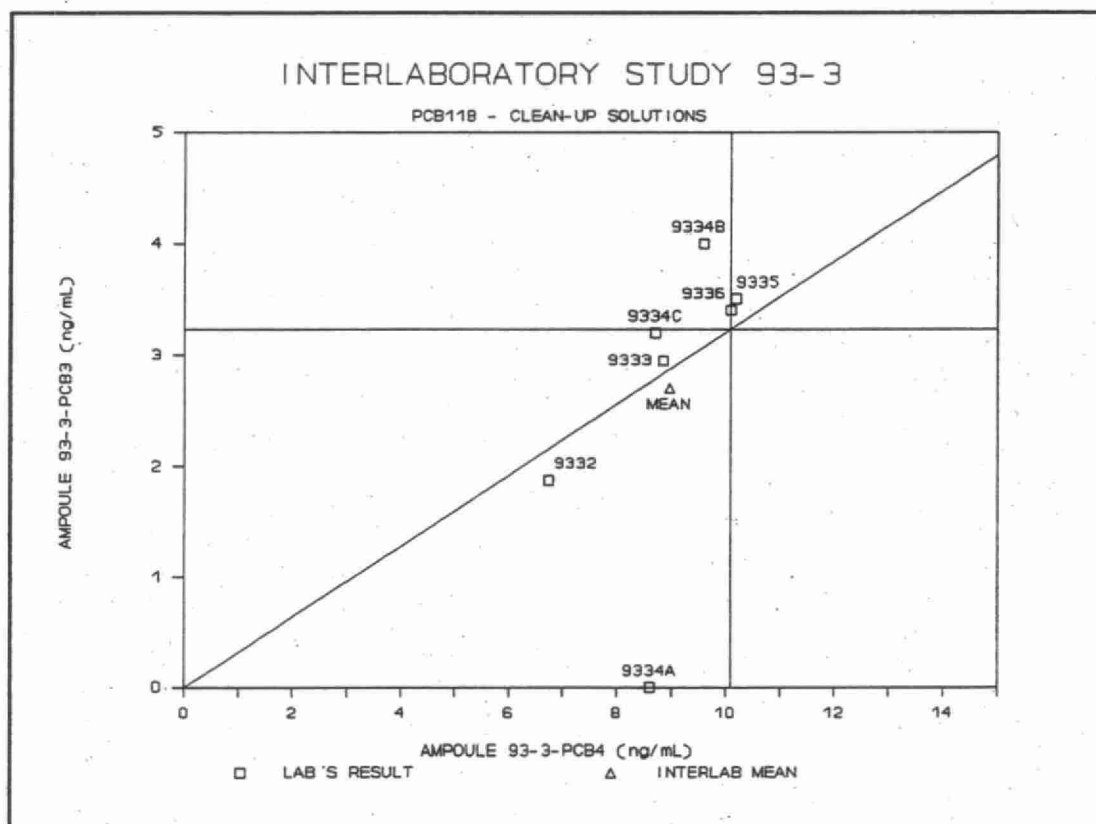


Figure 44 - PCB118 from Ampoules Processed through Clean-up

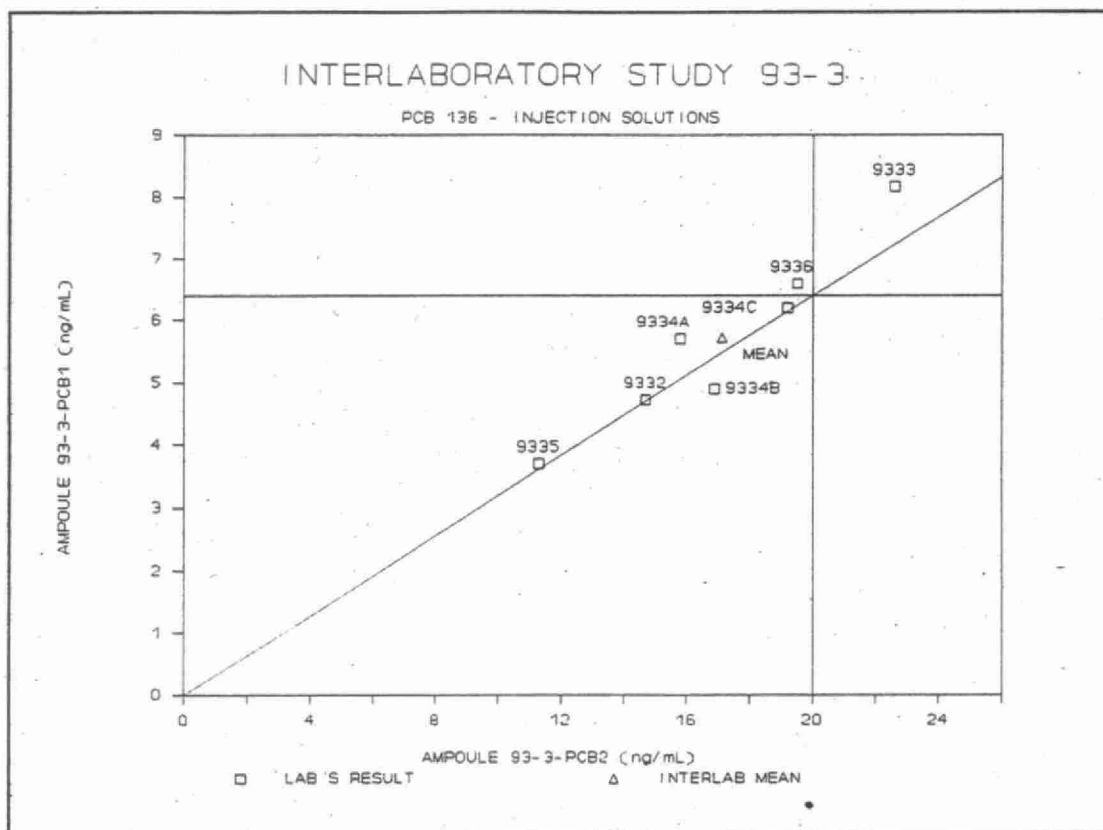


Figure 45 - PCB136 from Injection-Ready Ampoules

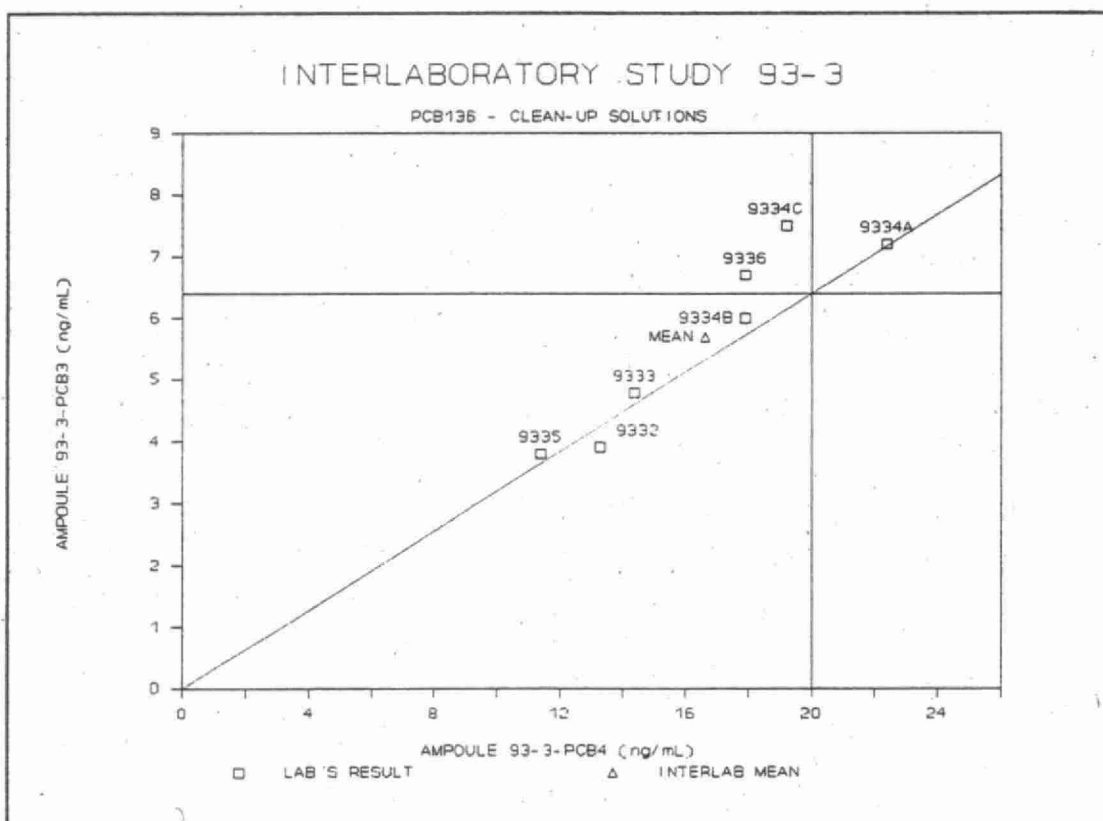


Figure 46 - PCB136 from Ampoules Processed through Clean-up

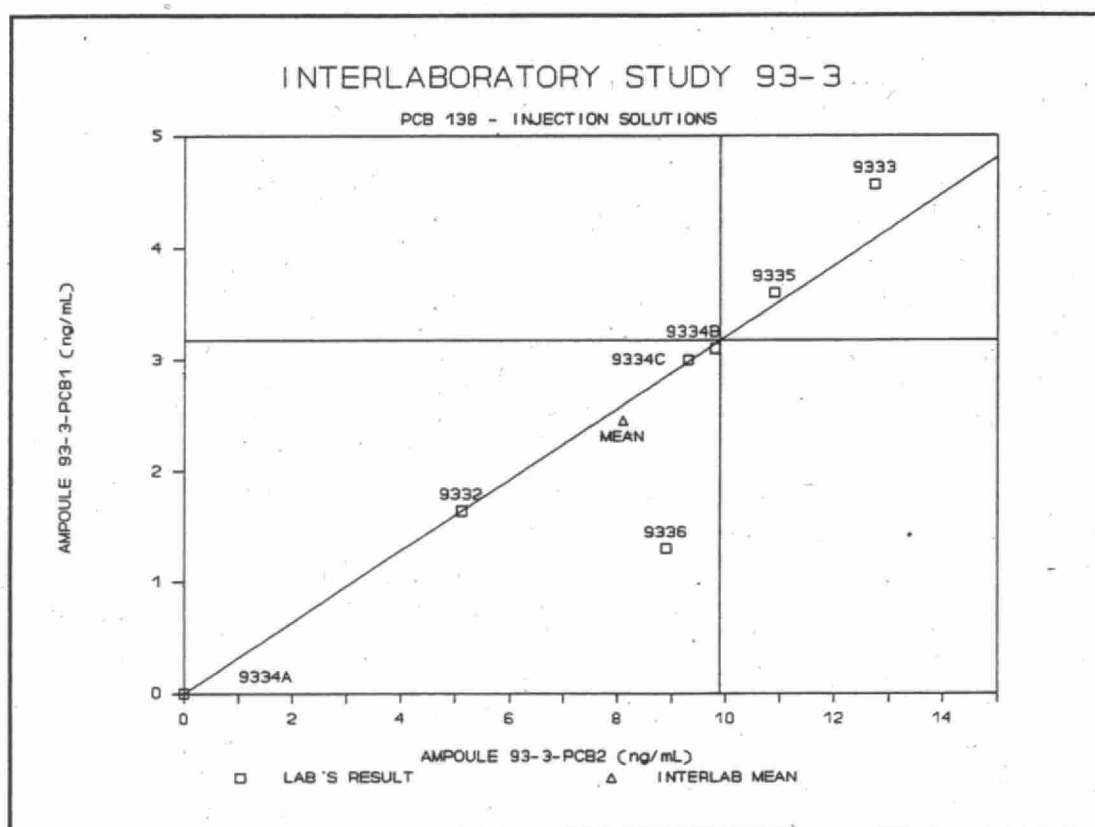


Figure 47 - PCB138 from Injection-Ready Ampoules

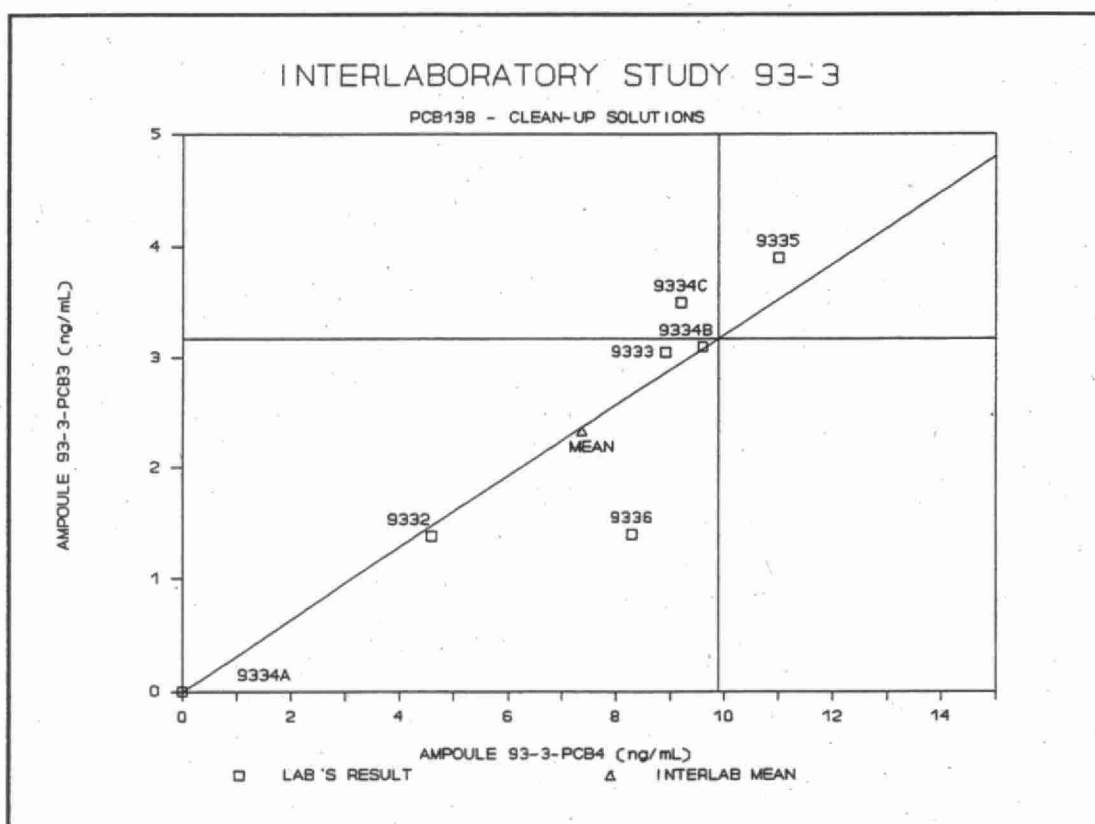


Figure 48 - PCB138 from Ampoules Processed through Clean-up

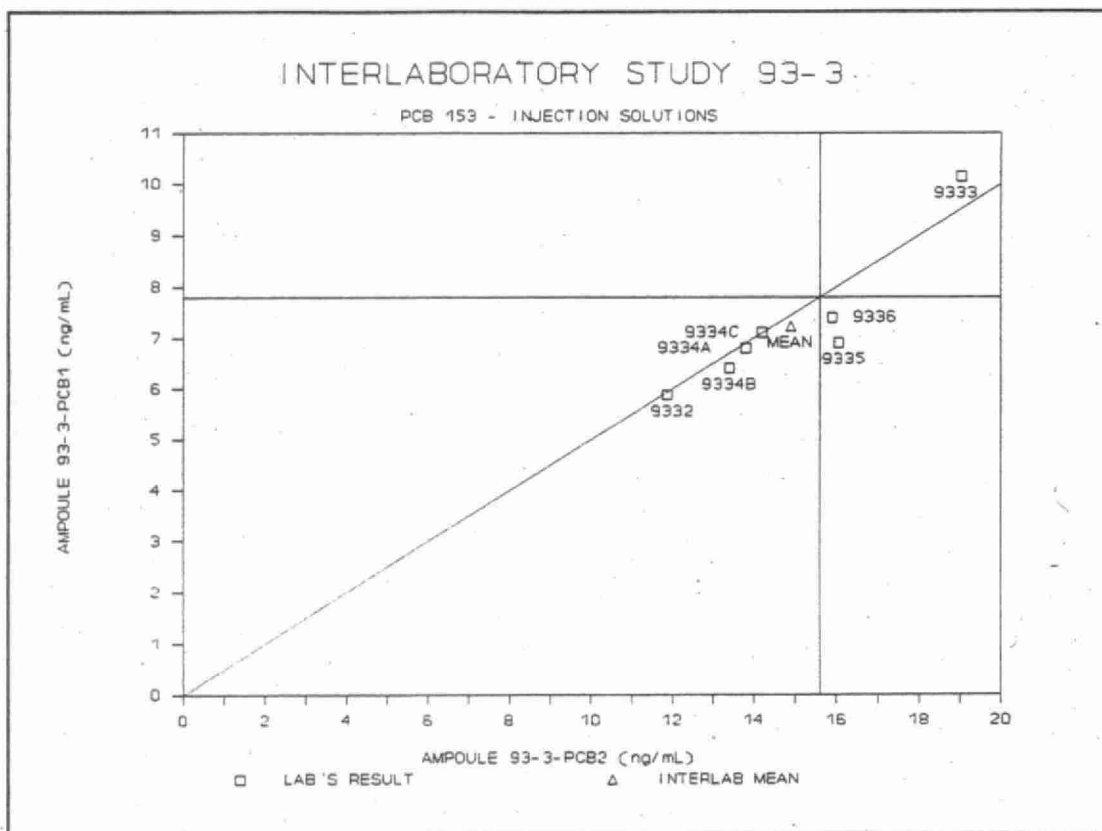


Figure 49 - PCB153 from Injection-Ready Ampoules

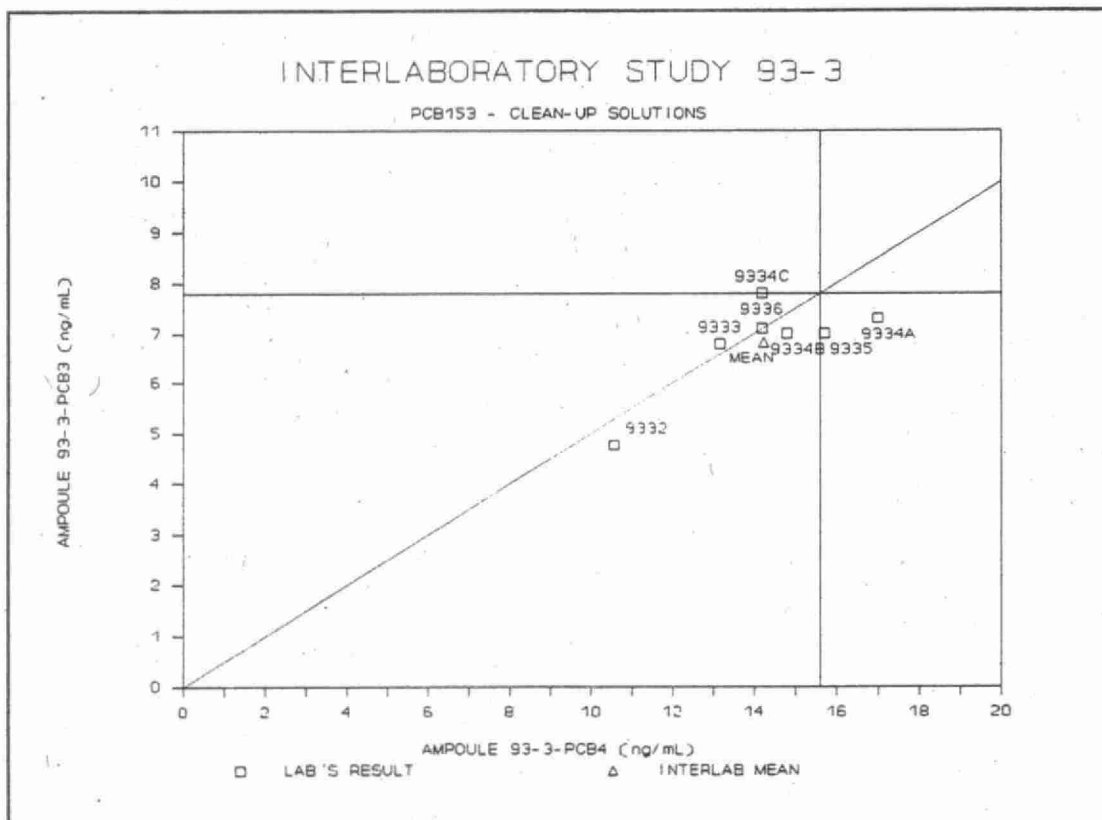


Figure 50 - PCB153 from Ampoules Processed through Clean-up

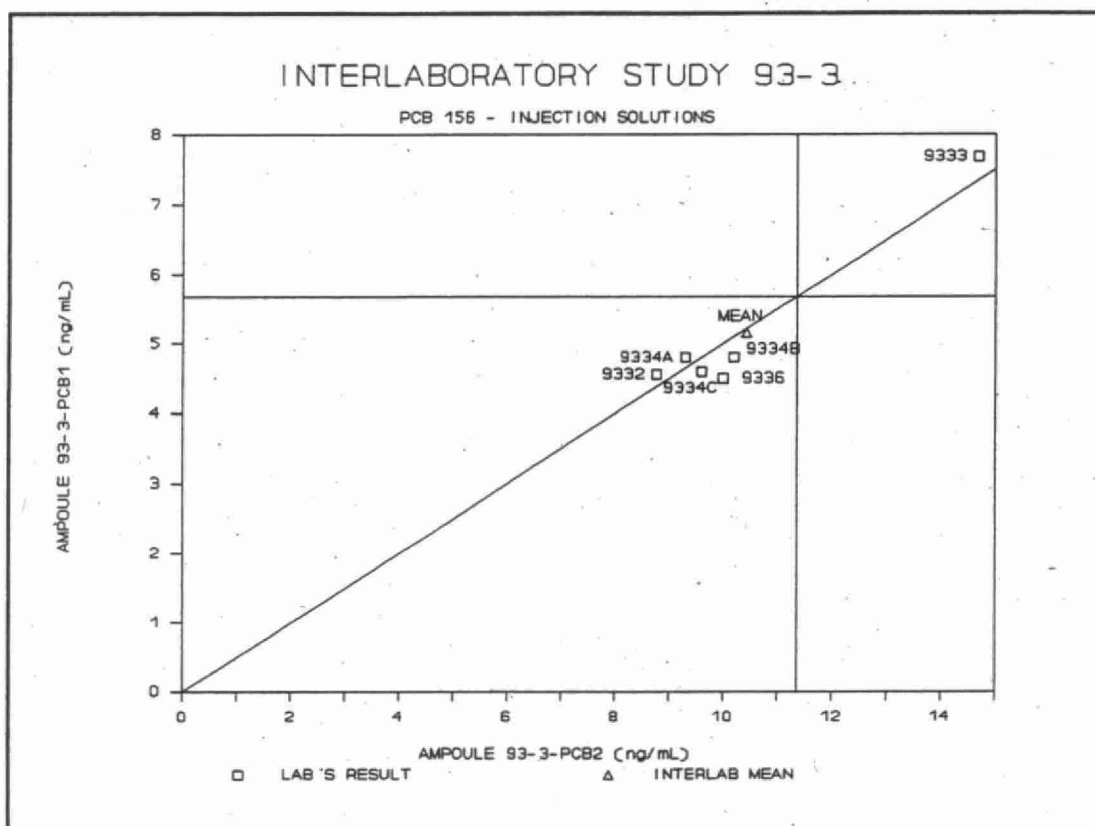


Figure 51 - PCB156 from Injection-Ready Solutions

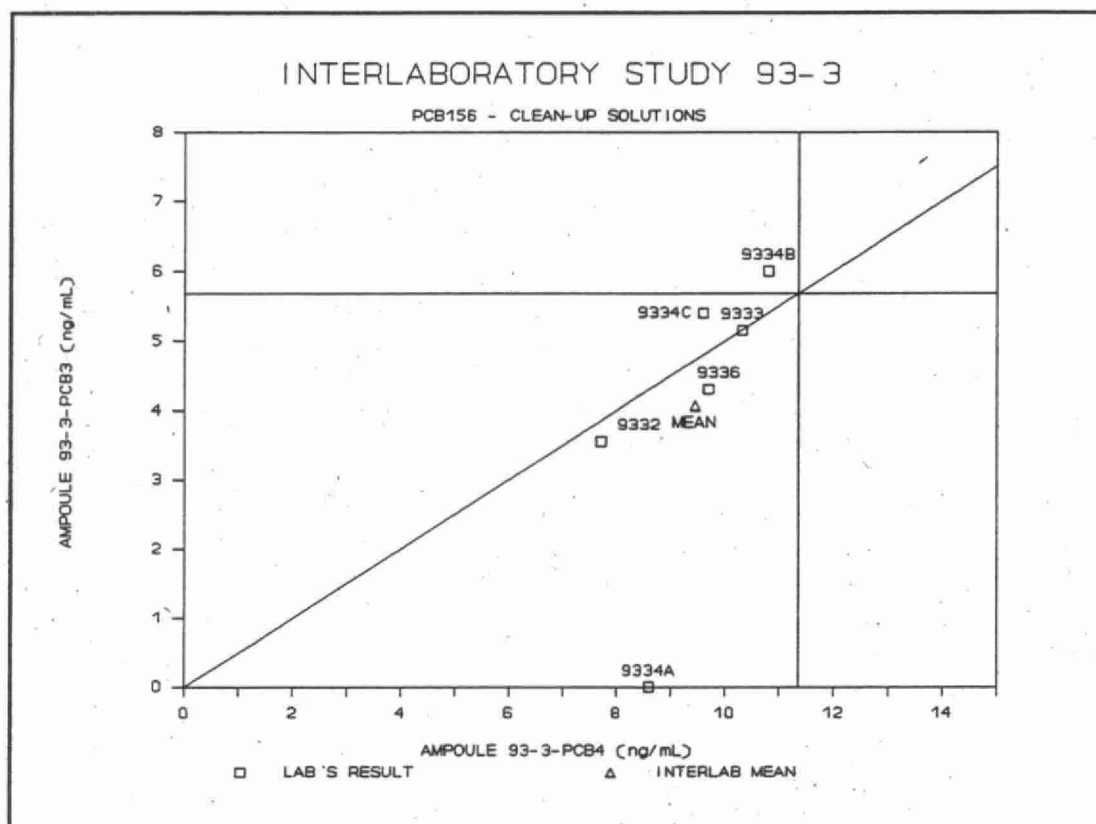


Figure 52 - PCB156 from Ampoules Processed through Clean-up

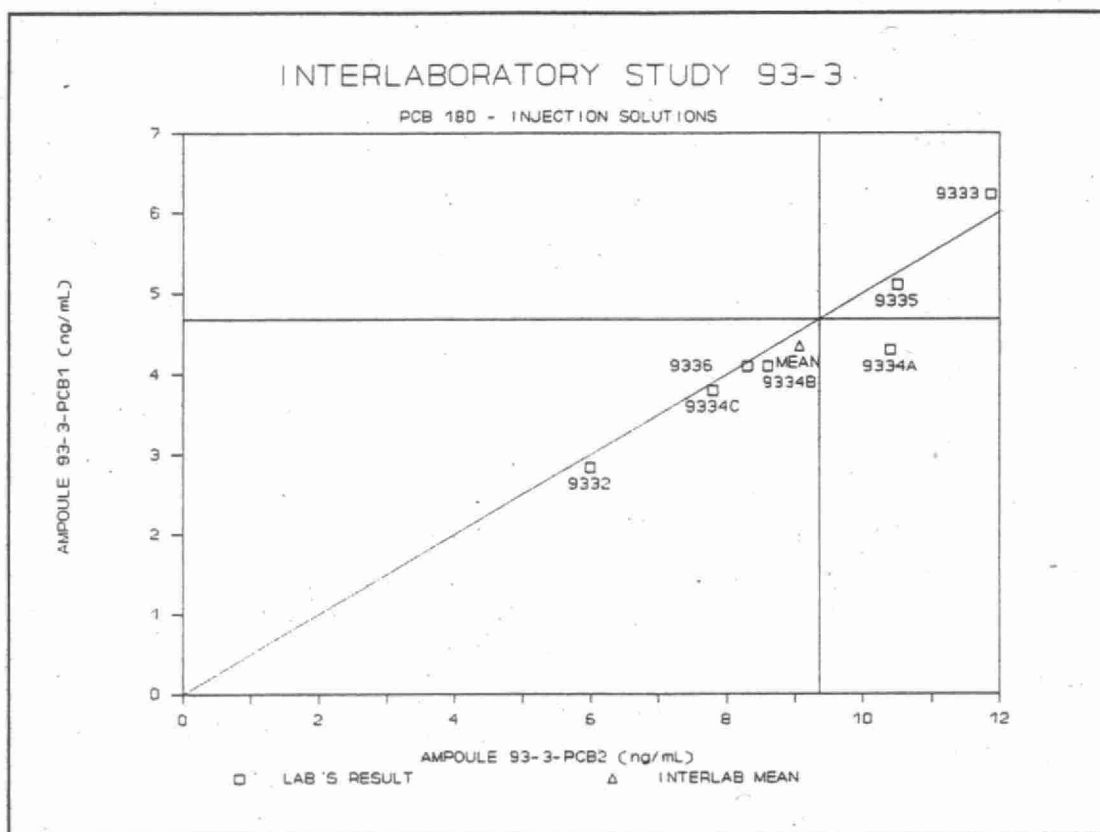


Figure 53 - PCB180 from Injection-Ready Ampoules

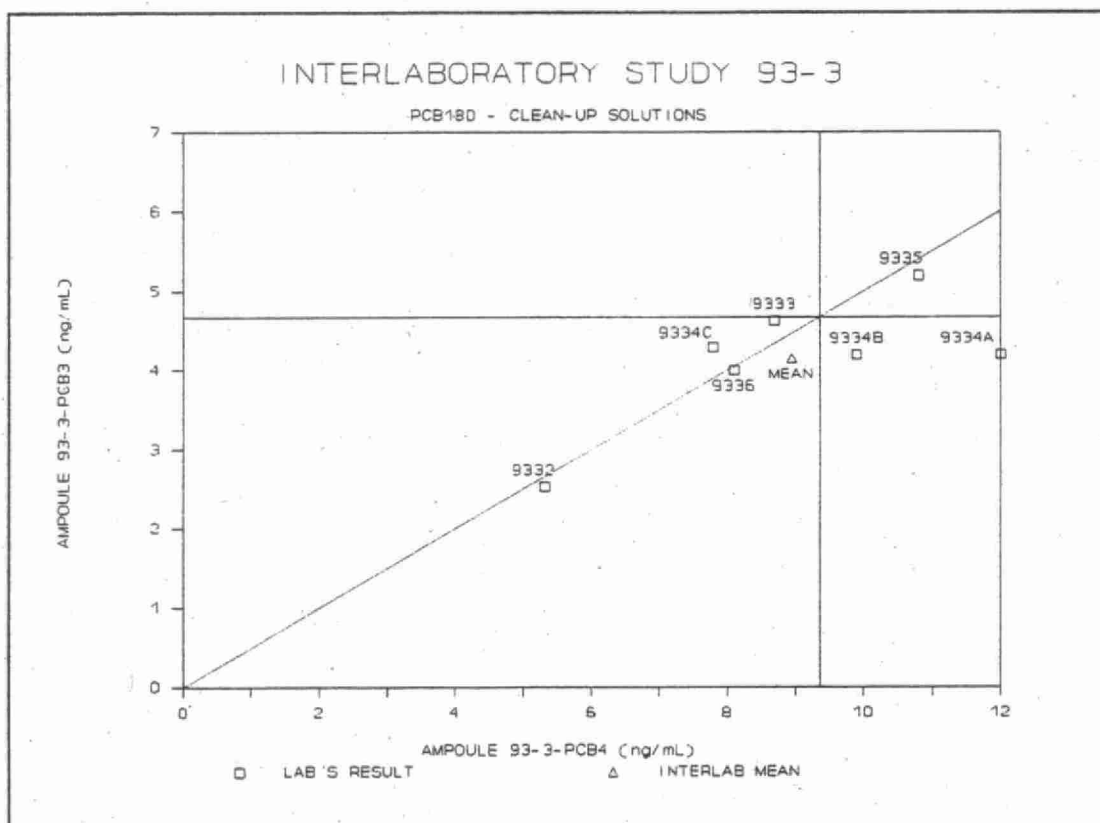


Figure 54 - PCB180 from Ampoules Processed through Clean-up

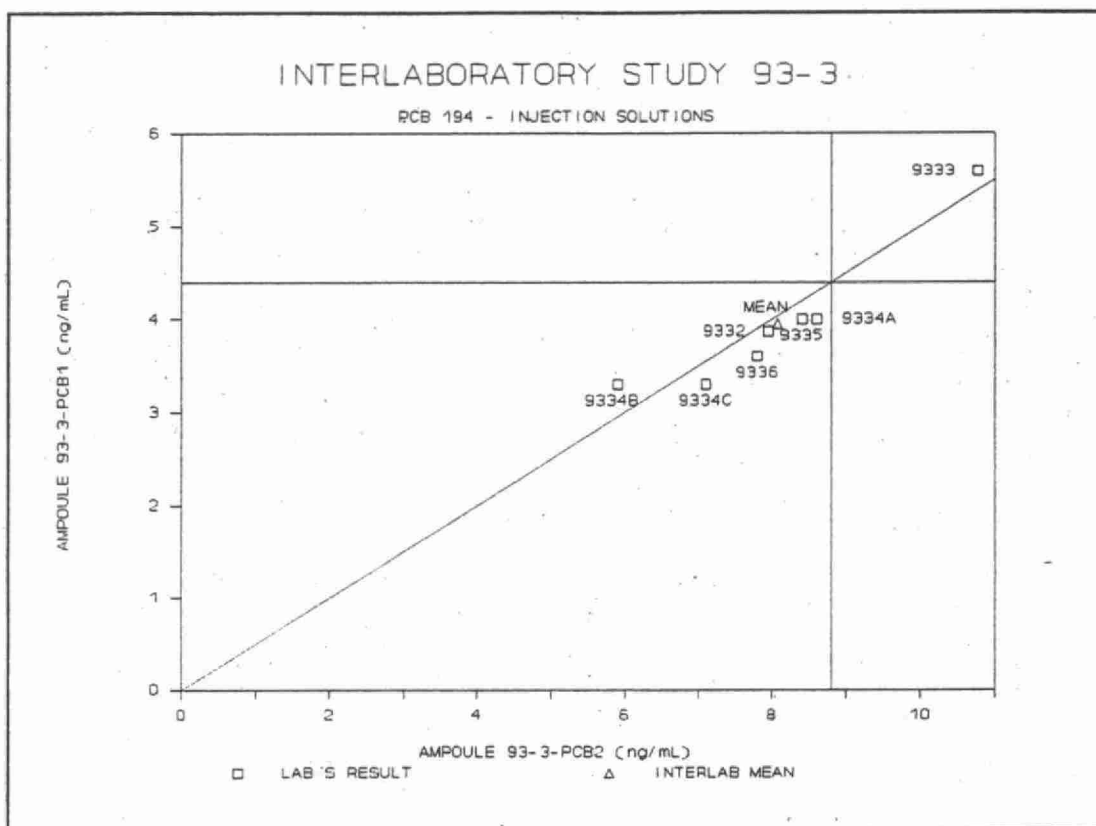


Figure 55 - PCB194 from Injection-Ready Ampoules

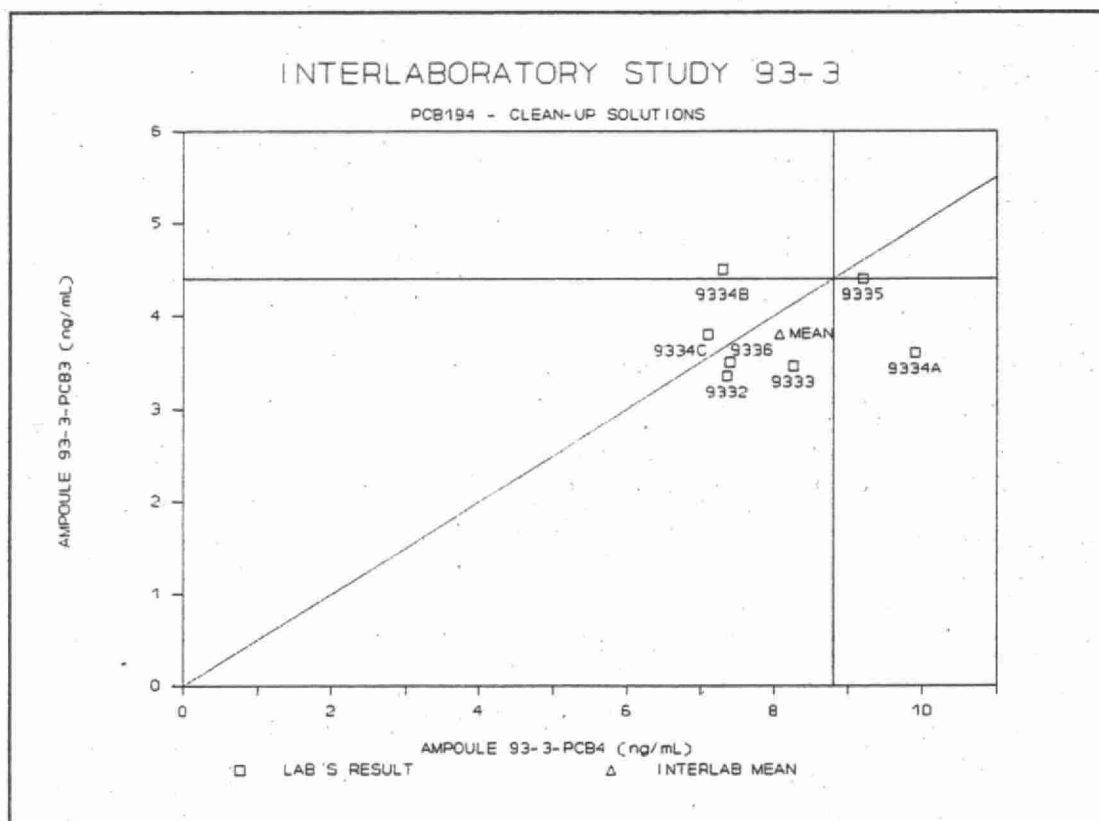


Figure 56 - PCB194 from Ampoules Processed through Clean-up

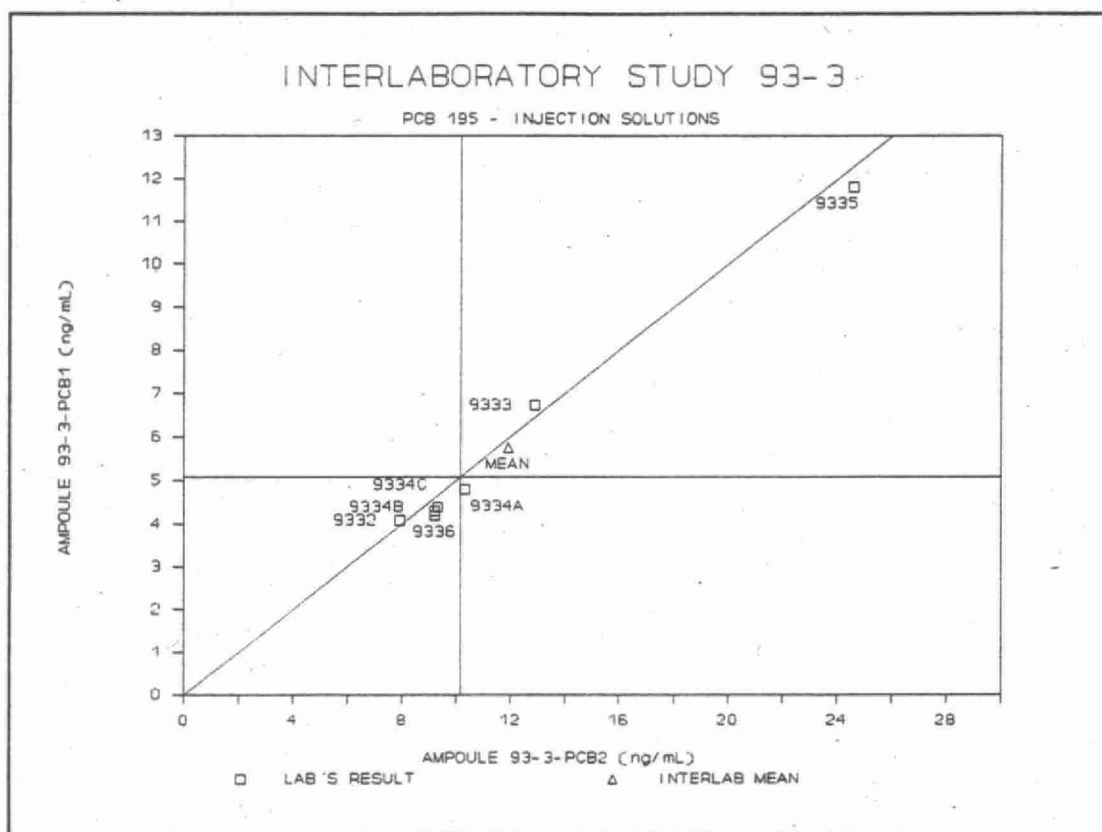


Figure 57 - PCB195 from Injection-Ready Ampoules

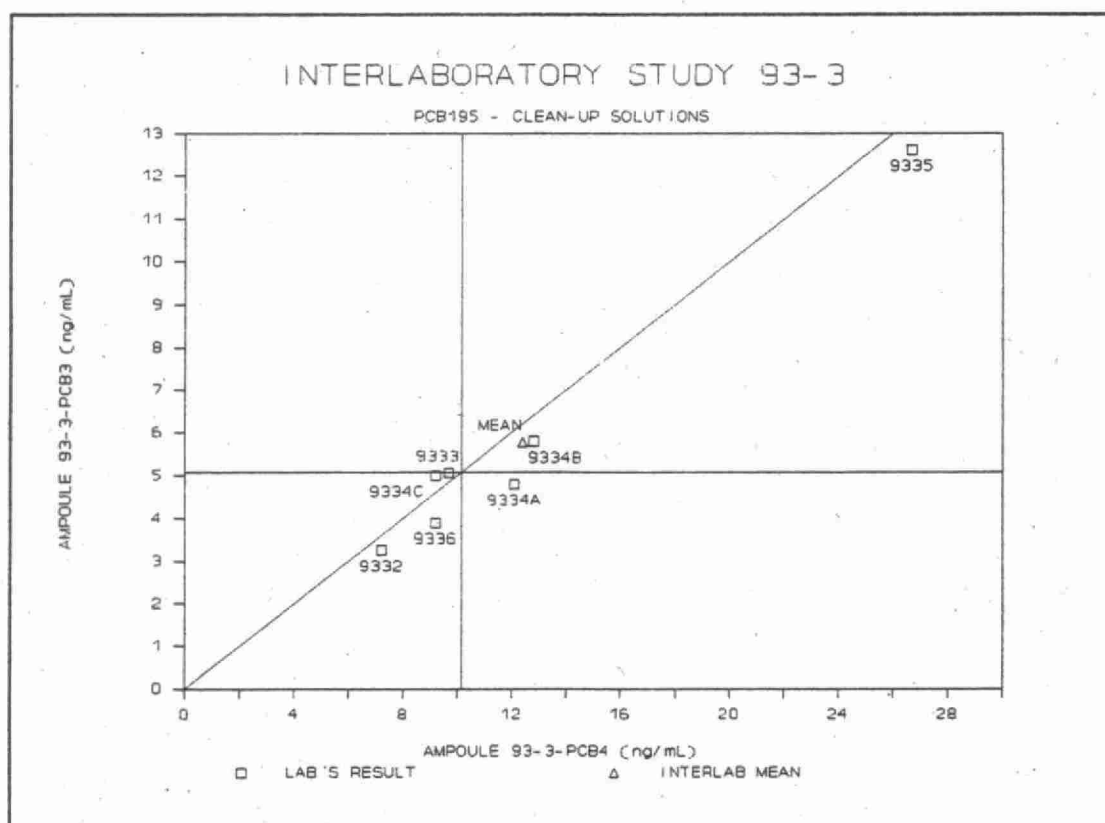


Figure 58 - PCB195 from Ampoules Processed through Clean-up

INTERLABORATORY STUDY 93-3

Distribution of Participants' Results

Injection-Ready Ampoules (PCB1 & PCB2)

Distribution of Results

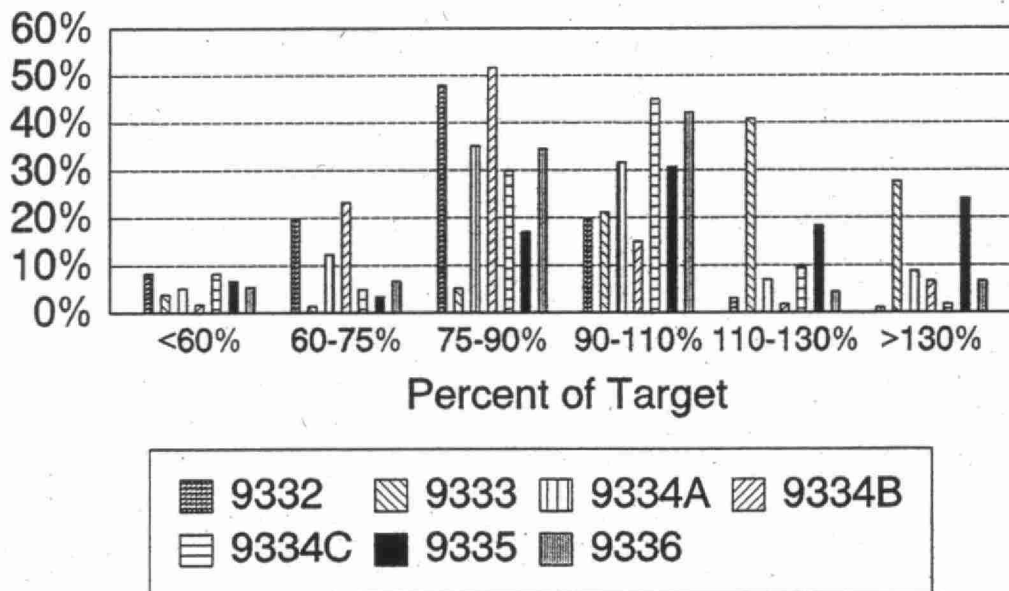


Figure 59 - Distribution of Participants' Results Relative to Target (PCB1 & PCB2)

INTERLABORATORY STUDY 93-3

Distribution of Participants' Results

Ampoules for Analytical Clean-up (PCB3 & PCB4)

Distribution of Results

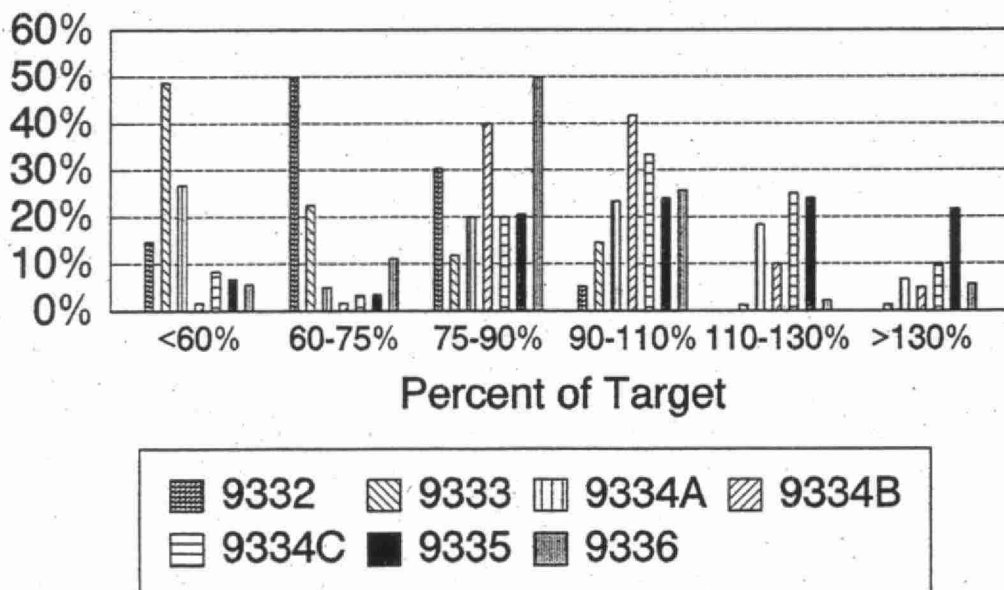


Figure 60 - Distribution of Participants' Results Relative to Target (PCB3 & PCB4)

8 APPENDIX 2 - PARTICIPANTS AND CORRESPONDENCE

List of Participants

William Strachan/Debbie Burniston
Lakes Research Branch
National Water Research Institute
867 Lakeshore Rd., P.O. Box 5050
Burlington, Ontario
L7R 4A6
(905) 336-4775/6025

Dan Toner/Paul Yang
Ministry of Environment and Energy
Laboratory Services Branch
Atmospheric & Biomaterials Analyses Section
125 Resources Rd.
Etobicoke, Ontario
M9P 3V6
(416) 235-5755/6004

Bert Grift
Department of Fisheries and Oceans
Freshwater Institute
501 University Cres.
Winnipeg, Manitoba
R3T 2N6
(204) 983-5167

Ilora Basu/Karen Harlin
Chemistry Division
Illinois State Water Survey
2204 Griffith Drive
Champaign, Illinois, U.S.A.
61820-7495
(217) 333-3712/6413

Ken Brice
Atmospheric Environment Service
Air Quality Process Research Division
4905 Dufferin St.
Downsview, Ontario
M3H 5T4
(416) 739-4601

Michel Bertrand
Dpt. de Chimie
CP 6128, Succ. A
Université de Montréal
Montréal, Quebec
H3C 3J7
(514) 343-6111, ext. 7555

ARQP
4905 Dufferin St.
Downsview, Ontario
M3H 5T4
(416) 739-4847

August 17, 1993

Dear Interlaboratory Study 93-3 Participant,

Please find enclosed four 5 mL ampoules for the analysis of Polychlorinated Biphenyl (PCB) Isomers. The ampoules are labelled 93-3-PCB1, 93-3-PCB2, 93-3-PCB3, and 93-3-PCB4 and all are in iso-octane. If you are missing any of the ampoules or they have broken in transit, please contact Sathi Selliah at (416) 235-5700 immediately for replacement. Please read the following instructions carefully. Please store all solutions below 0°C.

Ampoules 93-3-PCB1 and 93-3-PCB2

The ampoules are ready for direct instrumental analysis. Break open the ampoule on the scored mark and transfer the contents to the appropriate sample container for your analytical system. No dilutions should be required, but if you do so, please mark the dilution factor used on the accompanying report form.

Ampoules 93-3-PCB3 and 93-3-PCB4

These solutions require analytical clean-up prior to instrumental analysis. Please note that there is an interferant present that must be removed by the clean-up process. Please provide all the indicated information on the accompanying report form.

Please report all results on the accompanying form by October 8, 1993.

Thank you for your participation in this study. Please contact me if you have any questions or problems.

Your identification code is:

Sylvia Cussion
Air Toxics Quality Assurance Officer
(416) 739-4847
FAX (416) 739-5708

INTERLABORATORY STUDY 93-3

POLYCHLORINATED BIPHENYL (PCB) ISOMERS
FOR THE INTEGRATED ATMOSPHERIC DEPOSITION NETWORK

Identification Code:

Units:

	93-3-PCB1		93-3-PCB2	
PARAMETER	Dil. Factor	Result	Dil. Factor	Result
PCB4				
PCB5				
PCB8				
PCB15				
PCB16				
PCB17				
PCB18				
PCB22				
PCB28				
PCB31				
PCB32				
PCB33				
PCB37				
PCB40				
PCB41				
PCB42				
PCB44				
PCB47				
PCB48				
PCB49				
PCB52				
PCB53				
PCB61				
PCB66				
PCB70				
PCB71				
PCB74				
PCB75				
PCB77				
PCB81				
PCB84				
PCB95				

	93-3-PCB1		93-3-PCB2	
PARAMETER	Dil. Factor	Result	Dil. Factor	Result
PCB97				
PCB100				
PCB101				
PCB105				
PCB110				
PCB114				
PCB118				
PCB119				
PCB126				
PCB136				
PCB138				
PCB149				
PCB153				
PCB156				
PCB163				
PCB169				
PCB180				
PCB194				
PCB195				

Dil. Factor = Dilution Factor

INSTRUMENT AND DETECTOR USED FOR ANALYSIS:

GC COLUMN:

INTERLABORATORY STUDY 93-3

POLYCHLORINATED BIPHENYL (PCB) ISOMERS
FOR THE INTEGRATED ATMOSPHERIC DEPOSITION NETWORK

Identification Code:

Units:

	93-3-PCB3		93-3-PCB4	
PARAMETER	Dil. Factor	Result	Dil. Factor	Result
PCB4				
PCB5				
PCB8				
PCB15				
PCB16				
PCB17				
PCB18				
PCB22				
PCB28				
PCB31				
PCB32				
PCB33				
PCB37				
PCB40				
PCB41				
PCB42				
PCB44				
PCB47				
PCB48				
PCB49				
PCB52				
PCB53				
PCB61				
PCB66				
PCB70				
PCB71				
PCB74				
PCB75				
PCB77				
PCB81				
PCB84				
PCB95				

	93-3-PCB3		93-3-PCB4	
PARAMETER	Dil. Factor	Result	Dil. Factor	Result
PCB97				
PCB100				
PCB101				
PCB105				
PCB110				
PCB114				
PCB118				
PCB119				
PCB126				
PCB136				
PCB138				
PCB149				
PCB153				
PCB156				
PCB163				
PCB169				
PCB180				
PCB194				
PCB195				

Dil. Factor = Dilution Factor

SAMPLE VOLUME USED FOR CLEAN-UP:

SOLVENT(S) USED FOR CLEAN-UP:

COLUMN PACKING MATERIAL:

FINAL SAMPLE VOLUME:

ARQP
4905 Dufferin St.
Downsview, Ontario
M3H 5T4
(416) 739-4847
FAX (416) 739-5708

June 20, 1994

Dear Participant of Interlaboratory Study 93-3,

Please find enclosed the table of results from Interlaboratory Study 93-3, for the analysis of PCB Isomers in support of the Integrated Atmospheric Deposition Network. I apologize for the delay in reporting these results, but there was a delay in receiving results from one of the participants. If there are any transcription errors, please contact me at (416) 739-4847 by July 8, 1994.

A final report will be provided to all participants.

Your identification code is:

Sincerely,

Sylvia Cussion
Air Toxics Quality Assurance Officer
(416) 739-4847

LABORATORY LIBRARY



96936000119422



Environment Ontario

Laboratory Library
125 Resources Rd.
Etobicoke, Ontario M9P 3V6
Canada